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DEVELOPMENT OF COLLECTION METHODS FOR SEMIVOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR

by

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Cartridges containing solid adsorbents between layers of polyurethane foam (PUF) were evaluated for collecting chlorobenzenes, chlorophenols, hexachlorocyclohexanes (HCHs), and two-ring aromatic hydrocarbons. The 26-sq.cm cross-section glass cartridges, packed with Tenax (5-10 g), XAD-2 (14-16 g), or Florisil (30 g) between two 2.5-cm thick PUF slices, were designed to fit into the head of a General Metal Works PS-1 sampler. An inlet was built to vaporize test compounds into a clean airstream and sweep them into the adsorbent traps. Collection efficiency experiments at 20 deg C were conducted by injecting test compounds into the sampling train and determining by mass balance the percentages recovered from the traps. Retention volumes and theoretical plate numbers were determined for dichlorobenzene elution bands on PUF-Tenax cartridges. Dichlorobenzenes and two-ring aromatic hydrocarbons were collected from up to 165 cu. m air on PUF-Tenax (10 g Tenax), with mass balances averaging 88%. At 285-372 cu. m air, mass balances for tetra- through hexachlorobenzenes and HCHs averaged 89% on PUF-Tenax (5 g Tenax), 80% on PUF-XAD-2, and 78% on PUF-Florisil. Di- and trichlorophenols were collected without breakthrough by PUF-Tenax, but low recoveries were observed for pentachlorophenol and 2,3,4,5-tetrachlorophenol. Recoveries of chlorophenols were poorer from PUF-Florisil than from PUF-Tenax or PUF-XAD-2. Further chlorophenol collection studies are needed. Ambient air samples taken in urban and rural South Carolina demonstrated the utility of PUF-Tenax cartridges for collecting non-polar SOC.

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FOREWORD

Measurement and monitoring research efforts are designed to anticipate environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the ecology, to provide innovative means of monitoring compliance with regulations, and to evaluate the effectiveness of health and environmental protection efforts through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems for air, implementation of agency-wide quality assurance programs for air pollution measurement systems, and supplying technical support to other groups in the Agency, including the Office of Air and Radiation, the Office of Toxic Sustances, and the Office of Solid Waste.

The determination of toxic organic compounds in ambient air is an area of increasing importance to EPA. The primary goal of the program was to extend the capabilities of a commercially available high volume sampler for particulates and pesticides to low molecular semivolatile organic compounds. PUF-Tenax® "sandwich" sorbent cartridges were shown to be successful for the collection of low moleculr weight semivolatile organic compounds during 24 hr sampling periods.

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ABSTRACT

Cartridges containing solid adsorbents between layers of polyurethane foam (PUF) were evaluated for collecting chlorobenzenes, chlorophenols, hexachlorocyclohexanes (HCHs), and two-ring aromatic hydrocarbons. The 26-cm^2 cross-section glass cartridges, packed with Tenax (5-10 g), XAD-2 (14-16 g), or Florisil (30 g) held between two 2.5-cm thick PUF slices, were designed to fit into the head of a General Metal Works PS-1 air sampler. An inlet was built to vaporize test compounds into a clean airstream and sweep them into the adsorbent traps in the PS-1. Collection efficiency experiments at 20°C were conducted by injecting test compounds into the sampling train and determining by mass balance the percentages recovered from the traps. Retention volumes and theoretical plate numbers were determined from dichlorobenzene elution bands on PUF-Tenax cartridges.

Dichlorobenzenes and two-ring aromatic hydrocarbons were collected from up to 165 m^3 air on PUF-Tenax (10 g Tenax), with mass balances averaging 89%. At 285-372 m^3 air, mass balances for tetra- through hexachlorobenzenes and HCHs averaged 89% on PUF-Tenax (5 g Tenax), 80% on PUF-XAD-2, and 78% on PUF-Florisil. Di- and trichlorophenols were collected without breakthrough by PUF-Tenax, but low recoveries were observed for pentachlorophenol and 2,3,4,5-tetrachlorophenol. Recoveries of chlorophenols were poorer from PUF-Florisil than from PUF-Tenax or PUF-XAD-2. Further chlorophenol collection studies are needed. Ambient air samples taken in urban and rural South Carolina demonstrated the utility of PUF-Tenax cartridges for collecting SOC.

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ABBREVIATIONS

BIPH	Biphenyl
DCB	Dichlorobenzene
DCM	Dichloromethane
DCP	Dichlorophenol
DMN	Dimethylnaphthalene
EE	Diethyl ether
ECD	Electron capture detector (detection)
EI	Electron impact (mass spectrometry)
FID	Flame ionization detector (detection)
GC	Gas chromatography
HCB	Hexachlorobenzene
HCH	Hexachlorocyclohexane
Hi-vol	High volume
MID	Multiple ion detection (mass spectrometry)
MN	Methylnaphthalene
MS	Mass spectrometry
N	Theoretical plate number
NAP	Naphthalene
NI	Negative ionization (mass spectrometry)
p^o	Vapor pressure (phase unspecified)
p_L^o	Liquid-phase vapor pressure
p_S^o	Solid-phase vapor pressure
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PCP	Pentachlorophenol
PE	Petroleum ether
PeCB	Pentachlorobenzene

PUF	Polyurethane foam
SOC	Semivolatile organic compounds
TCB	Trichlorobenzene
TeCB	Tetrachlorobenzene
TeCB	Tetrachlorobenzene
TeCP	Tetrachlorophenol
TEO	Total extractable organic compounds
UCM	Unresolved complex mixture
V_B	Breakthrough volume
V_F	Retention volume at peak trailing edge
V_g	Retention volume per gram adsorbent
V_I	Retention volume at peak leading edge
V_R	Retention volume
V_S	Sampled air volume
$W_{1/2}$	Peak width at half height

SECTION 1

INTRODUCTION

Within the last decade solid adsorbents have become increasingly popular for preconcentrating trace organic compounds from air. Development of sampling methods has progressed along two main lines. Low volume (lo-vol) collection of volatile chemicals such as solvent vapors is done by drawing tens to hundreds of liters of air through columns containing a few hundred mg adsorbent. High volume (hi-vol) collection is generally used for "semivolatile" organic compounds (SOC), which have vapor pressures (p^o) roughly between $10^0 - 10^{-8}$ torr at ambient temperatures. Hi-vol samplers contain several grams adsorbent, and air volumes typically range from tens to thousands of cubic meters.

The lower volatility SOC ($p^o \leq 10^{-3}$ torr) include polychlorinated biphenyls (PCB), pesticides, 3-ring and higher polycyclic aromatic hydrocarbons (PAH), phthalate esters, polychlorinated dibenzo-p-dioxins (PCDD) and dibenzofurans (PCDF). Polyurethane foam (PUF) is a common adsorbent for hi-vol sampling of these SOC, however its utility is limited to SOC having vapor pressures below about 1.0E-3 torr (Bidleman, 1985).

The upper end of the range ($10^0 > p^o > 10^{-3}$ torr) includes some important pollutants for which air monitoring data are scarce: 2-ring PAH, chlorobenzenes, and chlorophenols. It is also this volatility range for which PUF is not effective as a hi-vol adsorbent. Lewis et al. (1982) showed that lower molecular weight PCB were poorly collected by PUF alone, but efficiently trapped using a "sandwich" cartridge in which a granular adsorbent such as Tenax-GC, XAD-2, or Florisil was contained between two PUF slices. Smaller sandwich cartridges using PUF-Tenax effectively retained chlorobenzenes in lo-vol collection studies (Lewis and MacLeod, 1982).

General Metal Works, Inc. (Village of Cleves, Ohio) has recently introduced the PS-1 hi-vol sampler for collecting particulate and vapor-phase pesticides (Figures 1 and 2). The sampling head consists of a 10-cm diameter filter holder backed up by an adsorbent cartridge. The standard 60-mm diameter x 125-mm long glass cartridge was designed to use PUF plugs, but can also be packed with granular adsorbents sandwiched between fine screens or PUF layers.

This study was carried out to evaluate the PS-1, loaded with PUF-adsorbent sandwich cartridges, for collecting chlorobenzenes, 2-ring PAH (naphthalenes and biphenyl) and chlorophenols. The granular adsorbent selected was Tenax-GC, although a few experiments were also carried out using XAD-2 resin and Florisil. Three types of studies were carried out:

1. Collection efficiencies for different SOC were determined in the laboratory by injecting known quantities of SOC into a prefiltered airstream, which was pulled through sandwich cartridges contained in the PS-1. The fraction collected was determined by a mass balance of the quantities injected and found on the cartridge.

2. Retention volumes (V_R) and theoretical plate numbers (N) were determined for dichlorobenzenes eluted through a sandwich cartridge under hi-vol airflow.

3. Ambient air samples were collected in the city of Columbia, SC and at North Inlet estuary on the South Carolina coast and analyzed for chlorobenzenes, 2-ring PAH, chlorophenols, and chlorinated pesticides.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

The use of sandwich cartridges of PUF-Tenax and PUF-XAD-2 as alternatives to PUF alone in the PS-1 high volume sampler can extend the collection capabilities to more volatile compounds. The breakthrough volume (V_B) of HCB ($p_L^o = 1.3 \times 10^{-3}$ torr) on a 7.6-cm diam. x 7.5-cm thick PUF plug is 125 m^3 (Burdick and Bidleman, 1981). This V_B is reduced to 73 m^3 on a 5.8-cm diam. x 7.5-cm thick plug in the PS-1, assuming proportionality between plug volume and V_B. By using 10 g Tenax between two PUF slices, SOC as volatile as dichlorobenzenes ($p_L^o = 1.1$-1.7 torr, 20°) or 2-ring PAH ($p_L^o \leq 0.2$ torr, 20°) can be quantitatively collected (avg. recovery = 95%, corrected for analytical losses) from up to 165 m^3 at 20°.

In a 24-h air sampling period (about 350 m^3 air), 1,2,4,5-tetrachlorobenzene (TeCB, $p_L^o = 5 \times 10^{-2}$ torr) and less volatile SOC can be trapped using 5-10 g Tenax between PUF slices. Under the same conditions, sandwich traps containing XAD-2 collected TeCB and heavier SOC, and traps containing 30 g Florisil collected pentachlorobenzene (PeCB) and heavier SOC. Average recoveries of TeCB and heavier SOC were higher from PUF-Tenax (89%) than from the other two sandwich cartridge types (80% on XAD-2, 78% on Florisil). These 24-h experiments were done using fairly low levels of chlorobenzenes and HCHs, and additional tests at higher loadings would be desirable.

Laboratory-determined retention volumes (V_R) of dichlorobenzenes on a PUF-Tenax sandwich (10 g Tenax) were in the 80-280 m^3 range at 20°. Collection efficiencies from laboratory and field experiments were consistent with V_R on the order of 200-250 m^3.

PUF-Tenax and PUF-XAD-2 cartridges that had been cleaned by solvent extraction, dried, and stored in an airtight container remained clean. Reanalysis of such cartridges after several months' storage gave generally clean blanks, with a few artifact peaks.

Some problems were encountered with operational blanks, in which analytically clean cartridges were subjected to a hi-vol airstream that had been passed through a glass fiber filter and then through a canister of activated carbon. Such blanks usually showed more contamination than analytical blanks, and in some cases higher levels of contamination were found on the back cartridge in the two-cartridge train. Further work needs to be done to determine the cause of this problem. Possibilities include: inefficient performance of the activated carbon prefilter, artifact formation on the adsorbents as a result of oxidation or resin "cracking", and momentary back-flow from the air pump to the cartridge canister. Until we understand why operational blanks are higher than analytical blanks, we cannot decide which blanks should be used to correct ambient air data.

Di- and trichlorophenols were efficiently collected from 285-358 m^3 air using PUF-Tenax sandwiches. Recoveries were $\geq$ 73% in five experiments; however two runs showed lower results. Pentachlorophenol gave very low recoveries in air sampling experiments (33% average). Similar results were obtained with PUF-XAD-2 sandwiches, but yields of chlorophenols using PUF-Florisil were substantially lower, even for the di- and trichlorophenols. Further work is needed with the chlorophenols to: a) determine the causes of PCP loss, b) include 2,4,5-TCP, an important chlorophenol not used in the present study, and c) test the collection system at varied chlorophenol loadings.

Ambient air samples taken at an urban and rural site confirmed the ability of PUF-Tenax cartridges to collect 2-ring PAH, n-alkanes $\leq C_{12}$, and chlorobenzenes from up to 350 m^3 air (24 h) without breakthrough losses. Heavier organochlorines such as PCB and chlordane were also collected. Concentrations of SOC determined by PUF-Tenax sampling were within the range of reported urban air values.

Analysis of "fraction 2" organochlorines (chlordanes, toxaphene) was hampered by an unresolved complex mixture (UCM) which was extracted from the front trap in a PUF-Tenax tandem cartridge train. This UCM was also noted in several (but not all) laboratory collection experiments and field samples taken with PUF-Tenax. The origin of the UCM is unknown, although there are indications that atmospheric oxidants may play a role in its formation.

SECTION 3

DETERMINATION OF VAPOR COLLECTION EFFICIENCIES.

The most direct way to evaluate the trapping ability of an adsorbent cartridge is to introduce known quantities of SOC into the airstream that is pulled through the cartridge. At the end of the experiment, SOC quantities found on the cartridge can be compared to those added. To most closely mimic an actual air sampling situation, the SOC vapors should be bled into the system continuously over the run (frontal mode). However it is experimentally easier to inject a single pulse of vapor at the beginning of the experiment (elution mode). Collection efficiencies of sandwich cartridges were evaluated in the latter mode, using multiple traps to follow the progress of the sample pulse through the train.

EXPERIMENTAL

Sampling Train Construction and Procedure for Collection Efficiency Experiments

The apparatus in Figure 3 was built to introduce test compounds into a clean airstream for sampling by the PS-1. Laboratory air was passed through a 20 x 25-cm glass fiber filter (Gelman A/E, binderless) to remove particles and then through a 77 mm diameter x 65 mm thick bed of activated carbon (6-14 mesh, Fisher Scientific Co.) to remove organic vapors. Methanol solutions (25-50 uL) of SOC were injected in a single pulse through a silicone rubber septum into a glass port. The compounds were vaporized and transported into a 12 x 15 x 100-cm stainless steel mixing chamber by passing precleaned air through the inlet while the glass bulb was warmed with heating tape. Air was pulled through the apparatus at 0.1 - 0.3 m^3/min, sweeping the SOC vapors from the mixing chamber into the adsorbent trap. Flow rates were determined by measuring the pressure drop with the Magnehelic gauge on the PS-1 (Figure 1). This pressure drop was related to flow rate using the PS-1 orifice calibrator. All collection experiments were carried out in a room thermostated at $20^\circ \pm 1^\circ$C.

Preparation and Cleanup of Solid Adsorbents

Polyurethane foam, product 3014 (density 0.022 g/cm^3), was obtained from Olympic Products Corporation, Greensboro, NC. Slightly oversized plugs were cut from 7.6-cm thick foam stock with a 2.75-inch (70 mm) hole saw using the technique of Simon and Bidleman (1979). These plugs were then cut into 2- or 3-cm thick slices using a band saw. PUF plugs were washed with tap water followed by reagent grade acetone. The plugs were then Soxhlet extracted for 24 h with reagent grade acetone followed by 24 h with pesticide quality petroleum ether (PE). After squeezing out the residual solvent, the plugs were dried in a vacuum desiccator at 40°C under constant water aspiration. Clean plugs were stored in mason jars with clean aluminum foil between the jar and lid.

Two types of Tenax resins were tested, Tenax-GC and the specially purified product Tenax-TA. Both were 35/60 mesh and were purchased from Alltech Associates, Deerfield, IL. Tenax was sieved with a 60-mesh screen to remove fines. In the early stages of the project, Tenax was cleaned up by soxhlet extraction in paper thimbles with PE for 24 h. After we discovered that chlorophenols were incompletely recovered from Tenax with PE alone, 15% ethyl ether (EE) in PE was used. Clean Tenax was vacuum-dried and stored as for PUF plugs.

XAD-2 (20-50 mesh) was obtained from Mallinckrodt Corporation. The resin was washed with tap water, and then soxhlet extracted for 24-h periods with the following solvents in the order listed: distilled water, dichloromethane (DCM), and 15% EE-PE. Clean XAD-2 was dried and stored as for PUF plugs.

Florisil (PR grade, 16-30 mesh), obtained from Floridan Corporation, was cleaned by baking in a muffle furnace overnight at 400°C. Clean Florisil was stored in mason jars as for PUF plugs.

Design of Adsorbent Cartridges

Several types of adsorbent cartridges were used over the course of this project:

1. Stacked Sieves. Experiments were run using 4 stacked glass sieves, each containing 3.9 g Tenax held on a thin PUF slice which was supported by a 10-mesh stainless steel screen (Figure 4). The sieve assembly was contained in an aluminum canister for attachment to the mixing chamber and hi-vol pump. The canister was our own design and not a stock PS-1 part. After sampling, the Tenax was transferred to paper thimbles for soxhlet extraction.
2. Double Sandwich Cartridge. Experiments were run with a standard PS-1 glass cartridge packed in a double-layer sandwich configuration (Figure 5). Tenax resin beds of 10 g (top trap) and 3 g (bottom trap) were separated and contained by

2-cm thick PUF slices; the bottom PUF slice was supported by a 10-mesh stainless steel screen. The filled cartridge was placed in a stock PS-1 aluminum canister, which connected the mixing chamber and hi-vol pump (Figure 3). After sampling, the cartridge was disassembled and the layers were individually soxhlet extracted.

3. Separate Front Sandwich and Back Traps. Experiments were run with the front and back traps in separate glass cartridges, held together by compression in the PS-1 aluminum canister, with a silicone rubber gasket providing a seal between the ground glass edges. The front trap contained 10 g Tenax sandwiched between two 2.5-cm PUF slices; the back trap contained 3 g Tenax on a 2.5-cm PUF slice with no top PUF layer (Figure 6). After sampling, the front trap was extracted intact in a specially designed soxhlet apparatus designed by the USC Chemistry Department Glassblowing Shop. This trap was dried, stored, and re-used without disassembly. Tenax from the back trap was transferred to a clean paper thimble for soxhlet extraction.

4. Separate Front and Back Sandwich Traps. Experiments were run using identical sandwich cartridges for front and back traps (Figure 7). Each cartridge contained Tenax, XAD-2, or Florisil between two 2.5-cm PUF slices. Different adsorbent weights were used depending on the experiment. An aluminum extender section was used between the PS1 filter holder and stock aluminum canister to hold the two cartridges. After sampling, cartridges were extracted intact and re-used without disassembly.

Test Compounds

Four classes of compounds were used to evaluate the collection efficiency of PUF-adsorbent sandwich cartridges:

1. Two-ring aromatic hydrocarbons: naphthalene (NAP), 2-methylnaphthalene (2-MN), 2,6-dimethylnaphthalene (2,6-DMN), and biphenyl (BIPH).
2. Chlorobenzenes: 1,2-dichlorobenzene (o-DCB), 1,3-dichlorobenzene (m-DCB), 1,4-dichlorobenzene (p-DCB), 1,2,4-trichlorobenzene (1,2,4-TCB), 1,2,4,5-tetrachlorobenzene (1,2,4,5-TeCB), pentachlorobenzene (PeCB), and hexachlorobenzene (HCB).
3. Hexachlorocyclohexanes (α- and γ-HCH).
4. Chlorophenols: 2,6-dichlorophenol (2,6-DCP), 2,4-dichlorophenol (2,4-DCP), 3,4-dichlorophenol (3,4-DCP), 2,4,6-trichlorophenol (2,4,6-TCP), 2,3,4,5-tetrachlorophenol (2,3,4,5-TeCP), and pentachlorophenol (PCP).

The HCHs, HCB, and PCP were analytical standards supplied by the U.S. Environmental Protection Agency Pesticides and Industrial Chemicals Repository, Research Triangle Park, NC. All other test compounds were purchased from chemical supply houses and used without further purification.

Vapor pressures of test compounds were in the $10^0 - 10^{-3}$ torr range at ambient temperatures. These are given as liquid-phase vapor pressures (p_L^o) in Table 1, since collection of SOC vapors on solid adsorbents appears to be controlled by the p_L^o rather than the solid-phase vapor pressure (p_S^o)(You and Bidleman, 1984; Bidleman et al., 1985). Interconversion of the two vapor pressures was made using (Mackay, 1982):

$$\ln P_L^o/P_S^o = 6.79\ (T_m - T)/T \qquad \text{Equation 1.}$$

In Equation 1, T_m and T are the melting point and ambient temperatures (Kelvin), and 6.79 is an average value for $\Delta S_f/R$, where ΔS_f is the entropy of fusion in cal/deg-mol and R is the gas constant (1.987 cal/deg-mol).

Analytical Procedures

Adsorbents in paper thimbles or PUF-adsorbent cartridges were soxhlet extracted overnight with PE or 15% EE-PE. Extracts were concentrated by flash evaporation to about 10 mL at room temperature, transferred to graduated centrifuge tubes, and then blown down to 5-10 mL in the presence of hexane or isooctane with a stream of nitrogen that had been filtered through clean Tenax.

Samples from laboratory collection experiments required less cleanup than ambient air samples because the air for these experiments was prefiltered through activated carbon. Analysis of two-ring aromatic hydrocarbons was done without further cleanup. Extracts to be analyzed for chlorobenzenes and HCHs were shaken with concentrated sulfuric acid before gas chromatographic (GC) analysis.

Chlorophenols were separated from base-neutral compounds and derivatized for GC analysis by the following procedure, adapted from Chau and Coburn (1974) and Abrahamsson and Xie (1983): The sample extract (2-5 mL) was shaken for 3.0 minutes with 5.0 mL of 0.1 M Na_2CO_3 in a 16 x 125 mm culture tube having a teflon-lined screw cap. The phases were separated, and the organic layer was saved for analysis of base-neutral compounds (e.g., chlorobenzenes and HCHs). The aqueous layer was shaken with 2 mL hexane and the hexane was added to the base-neutral fraction. One mL isooctane was added to the tube containing the aqueous phase, 50 μL acetic anhydride was added, and the tube was immediately capped and shaken for 3.0 min. This step derivatized the chlorophenols to their corresponding acetates and extracted them into the isooctane layer, which was then analyzed by GC.

GC analyses were carried out on a Carlo Erba 4160 chromatograph equipped with flame ionization (FID) and ^{63}Ni electron capture (ECD) detectors, and a Varian 3700 with a ^{63}Ni ECD. A 25-m BP-1 bonded phase (methyl silicone) fused silica column (SGE Corp.) was used for GC-FID work with aromatic hydrocarbons; columns used for organochlorine and chlorophenol analyses were 25-m fused silica bonded with 5% methyl phenyl silicone (BP-5, SGE Corp.; HP-5, Hewlett-Packard Corp.). Samples were injected splitless (Grob technique, 1-2 μL volume, 30 s split time). Several temperature programs were used over the course of this work and can be generalized as follows: Samples were injected with the column oven at 45-70°. After a 1-5 min hold, the oven was programmed at 5-8°/min to a final temperature of 220-265°. Other GC conditions were: carrier gas, helium or hydrogen at 20-40 cm/s; injector, 220-240°; detector, 320°. Chromatographic data were collected and processed with a Hewlett-Packard 3390 or Shimadzu Chromatopac CR3A integrator.

RESULTS AND DISCUSSION

Analytical Recovery of Test Compounds

Since the SOC used in this study are much more volatile than pesticides and PCB, greater care is required to minimize losses during sampling handling. All flash evaporations were done in a water bath at room temperature, and sample volumes were taken to only about 10 mL during this step. Further sample reduction was accomplished by nitrogen blow-down. Solvent evaporation during this step cooled the remaining sample and probably helped to retard solute loss.

Experiments were done with aromatic hydrocarbons and chlorobenzenes to determine losses that occurred during concentration steps. PE or pentane (100 mL) containing the solute quantities in Table 2 were concentrated by flash evaporation to about 15-20 mL, transferred to centrifuge tubes and blown down to 10 mL with nitrogen, and analyzed. The sample volumes were then further reduced with nitrogen to 1.0 mL and reanalyzed. A second series of similar experiments (Table 3) was done using 15% EE-PE, lower SOC quantities, and a final volume of 5.0 mL.

These recovery experiments are summarized in Tables 2 and 3. Recoveries of chlorobenzene were no better than about 75%, even when the final sample volume was 10 mL, and upon reduction to 1.0 mL the recovery dropped to 50% or lower. For this reason we decided not to work with chlorobenzene in collection efficiency studies. Recoveries of the less volatile chlorobenzenes and 2-ring aromatic hydrocarbons were 90% or better as long as the final volume was not reduced below 10 mL. Losses were greater if samples were concentrated to 1-5 mL. Some influence of the solvent was also seen; recoveries were better when PE alone rather than pentane was used (Table 2). Recoveries were lower for the experiments in Table 3. In these trials, lower SOC quantities were taken, the final volume was only 5 mL instead of 10 mL, and a different solvent (15% EE-PE) was used.

Adsorbents in paper thimbles or PUF-adsorbent cartridges were also spiked with SOC and extracted to determine recoveries for the entire analytical procedure. Chlorobenzenes and 2-ring aromatic hydrocarbons spiked onto Tenax in paper soxhlet thimbles were recovered to an extent of 84% or better using PE (Table 4). We initially tried PE to extract chlorophenols spiked onto Tenax, but recoveries were low and variable. Complete recovery was achieved with solvents of higher polarity however: 6% EE-PE, 15% EE-PE, and methanol (Table 5). A triplicate experiment with 15% EE-PE confirmed quantitative and reproducible recovery of chlorophenols (Table 6).

One experiment was done to check recoveries from a PUF-Tenax sandwich cartridge which was spiked with chlorobenzenes, HCHs, and chlorophenols. Recoveries of all compounds were 86% or better, with the exception of pentachlorophenol (Table 7).

Solid Adsorbent Blank Values

Two types of blanks were run during this project. Cleaned and stored adsorbents or PUF-adsorbent cartridges were soxhlet extracted and analyzed to give "analytical" blank values. Clean adsorbent cartridges were also placed in the PS-1, with the prefilter assembly in place, and air was drawn through the system to give "operational" blanks.

As received, Tenax-GC contained large quantities of total extractable organic residues (TEO)(Table 8). Most of this material was removed by 24-h soxhlet extraction with PE (Figure 8). Tenax thus cleaned remained clean, even after storage for 15-120 days in a tightly sealed container (mason jar) at room temperature (Tables 8 and 9; Figures 9 and 10). Tenax-TA received from the manufacturer contained far less TEO than did Tenax-GC, but after the initial cleanup its blank value was about the same as for Tenax-GC (Table 8). Considering the additional cost of Tenax-TA, and the fact that we planned to clean it up before use, there seemed to be no advantage to its use over Tenax-GC.

A common cleanup procedure for samples containing chlorinated hydrocarbon pesticides, PCB, and chlorinated benzenes is to shake a hexane or PE extract with concentrated H_2SO_4 or 7% fuming H_2SO_4. Many oxygen- and nitrogen-containing compounds are removed by this treatment, and chlorinated hydrocarbons are unaffected. PE or 15% EE-PE extracts of clean Tenax gave very "dirty" chromatograms by GC-ECD, but blanks were clean after acid treatment (Figures 11 and 12). However, the usefulness of Tenax for GC-ECD work may be limited to acid-stable chlorinated hydrocarbons.

The same solvent used for Tenax cleanup should be used for extracting the sample after collection. Tenax that had been used for several air sampling experiments and gave clean ECD chromatograms after extraction with PE was then re-extracted with 15% EE-PE. The resulting ECD chromatograms showed much extraneous material. It appeared that during air sampling polar residues were being built up (or formed) on the Tenax which were not extracted with PE, but released with 15% EE-PE.

Analytical blanks for PUF-Tenax cartridges, extracted with 15% EE-PE, are shown in Table 10. GC-ECD blanks (after acid cleanup) were clean, even after four months' storage, with the exception of o-DCB and 1,2,4-TCB (Figure 13). It is not known whether these residues are actually chlorobenzenes or other artifacts with the same retention times.

Naphthalene residues in XAD-2 extracts were a problem. In fact, we initially tried to use XAD-2 for cleaning up laboratory air via the prefilter assembly (Figure 3), but switched to activated carbon when it appeared that naphthalene was bleeding into the mixing chamber from the XAD-2 in the prefilter. Naphthalene as well as other aromatic hydrocarbons have been found as XAD-2 contaminants by Hunt et al. (1982, 1986). GC-ECD analytical blanks for PUF-XAD-2 cartridges showed artifacts that interfered with specific compounds, notably the dichlorobenzenes, 1,2,4-TCB, and 2,6-DCP. (Table 11).

Operational blanks were run by pulling air through the prefilter assembly and then through clean adsorbent traps. In some cases, these experiments were "dovetailed" with collection runs. For example, a chlorophenols collection experiment in which no chlorobenzenes were injected provided an operational blank for chlorobenzenes. Operational blanks were run to provide realistic background values for collection efficiency experiments. Such blanks for Tenax and PUF-Tenax are summarized in Tables 12-14, and for PUF-XAD-2 in Table 15. Examples of chromatograms are shown in Figures 14-16.

In general, operational blanks were higher than analytical blanks, especially for the more volatile compounds, and were higher when 15% EE-PE was used as an extracting solvent (cf. Tables 10 and 14, and Tables 11 and 15). The reason for this is not clear. Perhaps the prefilter was not 100% efficient, and SOC from laboratory air passed through the prefilter to contaminate the test cartridge. If this were the case, we would expect to find the highest operational blank residues on the front trap. As can be seen from Tables 12-15, this was sometimes (but not always) true. A second possibility is that air flowing through analytically clean adsorbent cartridges created artifacts (by surface oxidation?) or cracked the resins to expose uncleaned surfaces.

SOC quantities found on adsorbent cartridges from collection efficiency runs were corrected for average operational blank values before calculating recoveries. For high-level experiments, these blanks were only a small percent of SOC quantities taken. However the magnitude of the operational blanks was often similar to SOC quantities injected for dichlorobenzenes and 1,2,4-trichlorobenzene in low-level experiments. We are thus able to report recoveries for only 1,2,4,5-tetrachlorobenzene and heavier compounds for this group of experiments.

Possible Heavy Artifacts Formed During Sampling with Tenax

ECD chromatograms of PUF-Tenax cartridges that had been used for laboratory collection experiments or ambient air sampling sometimes showed a late-eluting unresolved complex mixture (UCM) that was resistant to sulfuric acid cleanup (Figures 17 and 18).

The UCM occurred in laboratory experiments even when a charcoal prefilter was used to provide "clean" air into the mixing chamber (Figure 3). The artifact was seen on front traps, only weakly or not at all on back traps, and was never observed with analytical Tenax or PUF-Tenax blanks. The UCM was also not observed in any PUF-XAD-2 collection efficiency runs or operational blanks. The problem thus seems to be related to Tenax.

We have been unable to find other studies in which this artifact was observed. However, most investigators using Tenax are not examining residues of heavy SOC, and the late-eluting UCM may never be observed. Many Tenax applications involve thermal desorption with an inert gas, and such a method may not desorb the heavy UCM contaminants.

We have not always seen this artifact. Between 1977-80 our laboratory collected many hi-vol ambient air samples using 10-g front and back Tenax traps (Billings and Bidleman, 1980, 1983), and never observed the UCM. Thinking that modern Tenax might have different properties from the Tenax in use during the late 1970's, we tested a sample of 1978-vintage Tenax (obtained from C. Sparacino, Research Triangle Institute). This Tenax also produced a UCM in a laboratory run with a charcoal prefilter in place.

The possibility that the UCM comes from SOC in laboratory air seems unlikely. Compounds with molecular weights as heavy as those in the UCM should be scrubbed by the charcoal prefilter. If heavy contaminants were passing through the prefilter, experiments with XAD-2 should have also shown the UCM.

It is possible that the artifact results from oxidation or depolymerization of Tenax when exposed to atmospheric oxidants such as O_3 or NO_x. Tenax breakdown while sampling air containing NO_x results in formation of 2,6-diphenylquinone and related products (Neher and Jones, 1977; Hanson et al., 1981), but these chromatograph as a few individual peaks, not as a UCM.

In an attempt to determine if atmospheric oxidants were responsible for producing the UCM, we carried out one collection efficiency experiment in which a layer of sodium thiosulfate (230 g) was placed in the prefilter on a slice of PUF above the charcoal layer. Filters impregnated with sodium thiosulfate have been used by other investigators to prevent Tenax oxidation and artifact formation (Harkov et al., 1985; Pellizzari et al., 1984). Neither the front nor the back PUF-Tenax trap showed a UCM in this experiment. Strangely however, subsequent runs in the laboratory without the sodium thiosulfate layer also failed to produce a UCM, and two ambient air samples taken at North Inlet estuary, SC showed no evidence of a UCM. We are thus unable to conclude that the use of sodium thiosulfate eliminated the artifact. In fact, its appearance and disappearance is still a mystery.

At this point we can only state that a UCM might be a problem with the use of Tenax for hi-vol sampling, and add this cautionary note for others. We have found that the UCM has the following analytical properties: a) It is observed only with ECD and not FID chromatograms. b) Treatment of sample extracts with concentrated sulfuric acid failed to eliminate the UCM. c) Silicic acid column chromatography is commonly used to fractionate organochlorines before GC-ECD analysis. In our procedure, PCB and chlorobenzenes are eluted in a non-polar fraction (PE) and most chlorinated pesticides (DDT, chlordane, HCH, endosulfan, dieldrin, toxaphene) are eluted in a polar fraction (DCM or PE-DCM). The UCM elutes in the latter fraction, and thus does not interfere with the analysis of PCB nor chlorobenzenes. It does interfere with the determination of late-eluting pesticides, particularly toxaphene, DDT, and to some extent with chlordane (Figures 17 and 18).

Collection Efficiencies for Test Compounds

The ability of different adsorbent cartridges to collect SOC vapors from 30-350 m^3 was tested by injecting known quantities of SOC into the mixing chamber of the sampling train (Figure 3), as described in the Experimental section, and determining the SOC recovered in multiple adsorbent traps. SOC quantities used in "high"- and "low"-level experiments are given in Table 16.

Aromatic hydrocarbon and organochlorine quantities found on adsorbent cartridges in collection experiments were corrected for average operational blanks before calculating recoveries. These blanks were generally lower for those experiments in which air volumes were $\leq 100\ m^3$ and cartridges were extracted with PE (Tables 12 and 13) than for experiments with volumes in the 288-368 m^3 range and 15% EE-PE extraction (Tables 14 and 15). Since we had only three of the latter blanks for PUF-Tenax, we included in the operational blank averages backup trap residues for tetrachlorobenzene and heavier organochlorines from some of the collection efficiency runs. Average operational blanks used to correct collection experiment recoveries are given in Tables 12 and 13 (air volumes $\leq$ 165 m^3, PE extraction) and Table 17 (air volumes $\leq$290 m^3, 15% EE-PE extraction).

Detailed results of the collection experiments are reported in the APPENDIX in two sets of A-tables. Sampling conditions, quantities of SOC taken, corrected quantities found on multiple traps, and recoveries are given for individual runs in the first set of tables. Recoveries and multiple trap distributions for different experiments are compared in a second set of spread sheets. Overall recoveries (sum of corrected quantities on multiple traps) using different adsorbent cartridges at varied air volumes are summarized in this section, Tables 18-26. Representative chromatograms are shown in Figures 20-22.

The first two experiments were carried out with stacked sieves containing 3.9 g Tenax and 100 m^3 air (Figure 4, Tables A1 and A2). For the most volatile aromatic hydrocarbon (NAP) and chlorobenzene (o-DCB) tested, 97% and 71% of the recovered quantities were found within the first two Tenax sieves.

The next nine experiments (Tables A3-A11) were done at air volumes in the 32-165 m^3 range, using double sandwich cartridges (Figure 5) or separate front sandwich and back Tenax traps (Figure 6). On the basis of the stacked sieve results, we decided upon 10 g Tenax for the front trap and 3 g for the back trap. Overall recoveries of 2-ring aromatic hydrocarbons at 34-165 m^3 were 74% or better in both high- and low-level experiments (Tables 18 and 19). NAP showed anomolously high recovery in one experiment, but the problem was traced to NAP contamination of XAD-2 resin that had been used in the prefilter for that experiment. Recoveries of 2,4,6-TCB and HCB in the same series of experiments were 77% or better (Tables 20 and 21), except for one stacked sieve run in which the 2,4,6-TCB recovery was only 67%.

Dichlorobenzene (DCB) results were more variable. In most experiments, 74-91% of the injected quantities were found. However, three runs gave yields of only 54-65%. Because of their volatilities (Table 2), DCBs are more prone to loss during analytical workup. When extracts were concentrated to about 5 mL, analytical yields of the DCBs were 84-87% (Tables 4 and 7), with one series of experiments giving lower recoveries (59-66%, Table 3). SOC recoveries in Tables 20 and 21 are uncorrected for analytical losses. If 85% analytical recovery is assumed for the DCBs, corrected overall recoveries of the DCBs in all collection experiments (Tables 20 and 21) are raised to 64-107% (average = 89%).

Breakthrough of 2-ring aromatic hydrocarbons and chlorobenzenes from front to back traps was low in this air volume range, as can be seen from Figure 19 and the summarized results in Table 27. A gradual increase in back trap residues with air volume can be seen for the DCBs, but at the maximum air volume used in these runs (165 m^3), only 11% of the DCBs were lost to the back trap. Breakthrough of the heavier aromatic hydrocarbons and chlorobenzenes was <2%.

Seven collection experiments were done using PUF-Tenax sandwich cartridges and air volumes in the 285-358 m^3 range. Two of these runs were made with 7 or 10 g Tenax in the front trap and 3 g in the back trap (Tables A13 and A28); the remaining experiments used 5 g Tenax in both front and back traps (Tables A14, A15, A23, A26, and A27). Chlorophenols were tested in all seven experiments; chlorobenzenes and hexachlorocyclohexanes were included in five of the runs.

Operational blanks for DCBs and 1,2,4-TCB were high compared to quantities injected in these collection experiments (Table 17), and we were therefore unable to determine recoveries for these compounds. Retention volumes at 20° for DCBs on a 10-g Tenax trap are 80-280 m^3 (Section III), and therefore the DCBs should not be quantitatively collected at the air volumes pulled in these experiments. Except for one experiment showing low results (Table A23), recoveries of 1,2,4,5-TeCB, PeCB, HCB, and γ-HCH were 76-112% (average = 93%). Yields of α-HCH were lower, ranging from 68-79% (average = 73%) (Table 21).

Organochlorine collection experiments using PUF-XAD-2 and PUF-Florisil are detailed in Tables A16-A22, A25, and A25, and summarized in Tables 22 and 23 in this section. Operational blanks for dichlorobenzenes were somewhat lower for PUF-XAD-2 than for PUF-Tenax (Tables 14, 15, and 17) and were lower than DCB quantities injected in PUF-XAD-2 collection experiments. Although not included in Table 22, the DCBs showed extensive breakthrough to back PUF-XAD-2 traps at 348-372 m^3 air and were not quantitatively collected (Tables A16-A18, A22). Operational blanks were not run for PUF-Florisil, but the sum of uncorrected front and back trap DCBs was far lower than quantities injected (Table 23). The yield of 2,4,6-TCB was also only 34-36% in two out of three experiments with PUF-Florisil. Considerable TCB broke through to the back trap, even for the one "good" 88% yield experiment (Tables A20 and A25).

Recoveries of heavier chlorobenzenes and HCHs in PUF-XAD-2 and PUF-Florisil experiments were complete in some experiments, but not in others (Tables 22 and 23). In the case of PeCB, HCB, and the HCHs, the average operational blanks included one result that was rather high, but nevertheless could not be excluded by a Q-test (Tables 15 and 17). If the high blank were excluded and the remaining four operational blanks averaged and used to correct collection experiment results, the recoveries of these compounds in Table 22 would be raised by about 5-10% (e.g., 48% instead of 38% for α-HCH, Table A18 and the second experiment in Table 22).

Collection of the heavier chlorobenzenes and HCHs using PUF-Tenax, PUF-XAD-2, and PUF-Florisil is summarized in Table 28. Average recoveries were 63% or better using any of the three systems, with PUF-Tenax apparently having the edge towards higher recoveries. It should be pointed out that we were "pushing" the lower limits of the method. Injected quantities of HCB and the HCHs in the low-level experiments ranged from 13-35 ng (Table 16). The concentration ranges (ng/m^3) for these SOC in continental air are 0.05-0.4 (HCB), 0.01-1.6 (γ-HCH), and 0.07-2.3 (α-HCH)(Section IV, Table 38). Quantities of these SOC taken in our experiments are, in some cases, near the lower end of the quantities that would be preconcentrated from 350 m^3 ambient air. Additional collection experiments using somewhat higher loadings of these SOC would be desirable.

Collection efficiencies of di- and trichlorophenols on PUF-Tenax were $\geq$73% in five out of seven experiments at air volumes in the 285-358 m^3 range (Tables A13-A15, A23, A26-A28; and Table 24 in this section). Some difficulty was encountered with 2,3,4,5-TeCP; two experiments that showed $\geq$90% recoveries for di- and trichlorophenols yielded only 65-66% of the injected TeCP. Poor recovery of PCP was observed in all cases. Analytical recoveries of PCP and other chlorophenols were quantitative in experiments in which Tenax contained in paper thimbles was spiked and extracted (Tables 5 and 6). Quantitative recovery of di- through tetrachlorophenols was also observed with a spiked PUF-Tenax cartridge, although in this case the yield of PCP was only 54% (Table 7). Poor results for PCP in collection experiments might be caused by difficulties in extracting vapor-deposited PCP from PUF-Tenax or to loss of PCP somewhere in the sampling train before the adsorbent cartridge. The somewhat lower recoveries of TeCP in two experiments showing good yields of the lighter chlorophenols suggests that TeCP may be subject to the same loss processes as for PCP, although to lesser extent.

In addition to low PCP recoveries, we observed some PCP blank problems. PUF plugs are cut with a hole saw, using a plywood template (Simon and Bidleman, 1979), and we identified PCP in sawdust from the template. A glass fiber filter that was not baked before use contained about 900 ng PCP, which may have come from contact with the cardboard in the filter box.

Two experiments showed unaccountably low recoveries of all chlorophenols (Tables A26, A27, and Table 24). Breakthrough of dichlorophenols to backup traps was $\leq$15%, and less for tri- and tetrachlorophenols, so breakthrough was not responsible for these losses. Analytical standards of chlorophenols were derivatized at the same time as samples. Incomplete sample derivatization in these two experiments could have caused the observed low recoveries, but there was no evidence that such a problem occurred.

Chlorophenols recoveries with the PUF-XAD-2 system showed problems similar to those encountered with PUF-Tenax. Less than 40% of the PCP could be accounted for in any experiment, and 2,3,4,5-TeCP gave low results in 3 out of five runs (Tables A16-A19, A22, Table 25). Dichlorophenol recoveries were 65-121% (average = 85 ± 17), except for one experiment in which yields of all chlorophenols was poor.

Chlorophenol recoveries using PUF-Florisil were lower than with either PUF-Tenax or PUF-XAD-2 (Tables A20, A21, A24, and Table 26). Even the dichlorophenols were poorly recovered. In some cases appreciable percentages of 2,6-DCP and 2,4-DCP were found on back traps, indicating that breakthrough may have been responsible for the low recoveries.

At this point, we cautiously suggest that PUF-Tenax can be used for quantitative collection of di- through trichlorophenols. However further collection experiments should be carried out to: a) determine whether the two runs showing low recoveries are spurious, b) determine the cause and cure for the occasionally low TeCP and consistently low PCP yields, and c) include 2,4,5-TCP, a common chlorophenol that was not used in the above experiments. Work on the above problems is now underway in our laboratory, supported by a subcontract from Battelle.

TABLE 1. Liquid-Phase Vapor Pressures of Organic Compounds.

	p_L^o, torr (20°C)[a]	Reference
Chlorobenzenes		
Chlorobenzene	9.32	Weast, 1971
1,3-Dichlorobenzene	1.69	Weast, 1971
1,4-Dichlorobenzene	1.45	Weast, 1971
1,2-Dichlorobenzene	1.06	Weast, 1971
1,2,4-Trichlorobenzene	0.32	Weast, 1971
1,2,4,5-Tetrachlorobenzene	0.052	Weast, 1971
Pentachlorobenzene	4.3×10^{-3}	Weast, 1971
Hexachlorobenzene	1.3×10^{-3}	Bidleman, 1984
Hexachlorocyclohexanes		
α-Hexachlorocyclohexane	6.3×10^{-4}	Balson, 1947
γ-Hexachlorocyclohexane	2.3×10^{-4}	Bidleman, 1984
Chlorophenols		
2,4-Dichlorophenol	0.10	Weast, 1971
2,6-Dichlorophenol	0.066	Weast, 1971
2,4,5-Trichlorophenol	0.050	Weast, 1971
2,4,6-Trichlorophenol	0.034	Weast, 1971
Pentachlorophenol	7.9×10^{-4}	Weast, 1971
Aromatic Hydrocarbons		
Naphthalene	0.20	Bidleman, 1984
2-Methylnaphthalene	0.048	Mackay, 1982
Biphenyl	0.024	Bidleman, 1984

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a) Literature solid-phase vapor pressures were converted to p_L^o using Equation 1. In some cases, vapor pressures at 20° were estimated from those given at 25° using heats of vaporization for compounds of similar volatility.

TABLE 2. Recovery of Test Compounds During Solvent Concentration Steps.

		% Recovery and Final Volume			
	Quantity	Petroleum Ether[a]		Pentane[b]	
Compound	µg	10 mL	1.0 mL	10 mL	1.0 mL
chlorobenzene	422	74	52	74	45
1,4-dichlorobenzene	123	99	72	91	61
1,2-dichlorobenzene	644	95	72	90	60
1,2,4-trichlorobenzene	433	95	77	90	62
naphthalene	136	98	78	88	63
2-methylnaphthalene	123	100	78	93	63
2,6-dimethylnaphthalene	109	100	80	93	69
biphenyl	139	101	80	94	65

a) average of 2 experiments. b) average of 3 experiments.

TABLE 3. Recovery of Test Compounds During Solvent Concentration Steps.

Compound	Quantity µg	% Recovery, 5-mL Volume 15% EE-PE[a]
1,2-dichlorobenzene	2.0	66
1,3-dichlorobenzene	2.7	59
1,4-dichlorobenzene	2.8	58
1,2,4-trichlorobenzene	0.42	70
1,2,4,5-tetrachlorobenzene	0.26	81
hexachlorobenzene	0.017	88

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a) average of three experiments.

TABLE 4. Recovery of Chlorobenzenes and Aromatic Hydrocarbons from Tenax Spiked in Paper Thimbles[a]

Compound	Quantity µg	Average % Recovery ± S.D.
1,2-dichlorobenzene	322	85 ± 3
1,4-dichlorobenzene	61	84 ± 3
1,2,4-trichlorobenzene	217	90 ± 2
naphthalene	68	90 ± 4
2-methylnaphthalene	61	88 ± 7
2,6-dimethylnaphthalene	54	90 ± 3
biphenyl	69	89 ± 3

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a) 3.9-g Tenax portions, three experiments, samples soxhlet extracted with PE.

TABLE 5. Recovery of Chlorophenols from Tenax Spiked in Paper Thimbles Using Different Solvents[a]

	% Recovery and Solvent		
Compound	6% EE-PE	15% EE-PE	Methanol
2,6-dichlorophenol	92	94	91
2,4-dichlorophenol	91	99	92
3,4-dichlorophenol	87	101	92
2,4,6-trichlorophenol	96	91	94
2,3,4,5-tetrachlorophenol	95	97	92
pentachlorophenol	96	100	98

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a) 3-g portions of Tenax. Chlorophenol quantities were 0.06-0.5 μg.

TABLE -6. Recovery of Chlorophenols from Tenax Spiked in Paper Thimbles Using 15% EE-PE[a].

Compound	Quantity µg	% Recovery ± S.D.
2,4-dichlorophenol	0.48	94 ± 5
2,6-dichlorophenol	0.51	91 ± 5
3,4-dichlorophenol	0.47	95 ± 12
2,4,6-trichlorophenol	0.23	93 ± 2
2,3,4,5-tetrachlorophenol	0.11	93 ± 3
pentachlorophenol	0.060	86 ± 4

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a) 3-g Tenax portions, three experiments.

TABLE 7. Recovery of Test Compounds from a Spiked PUF-Tenax Sandwich Cartridge[a].

Compound	Quantity µg	% Recovery
1,2-dichlorobenzene	2.1	87
1,3-dichlorobenzene	2.7	86
1,4-dichlorobenzene	2.9	90
1,2,4-trichlorobenzene	0.43	98
1,2,4,5-tetrachlorobenzene	0.27	96
pentachlorobenzene	0.029	103
hexachlorobenzene	0.017	105
α-hexachlorocyclohexane	0.024	96
γ-hexachlorocyclohexane	0.024	102
2,6-dichlorophenol	0.51	88
2,4-dichlorophenol	0.48	86
3,4-dichlorophenol	0.97	99
2,4,6-trichlorophenol	0.23	90
2,3,4,5-tetrachlorophenol	0.11	91
pentachlorophenol	0.060	54

---

a) 5 g Tenax between 2-cm thick PUF slices, extracted with 15% EE-PE.

TABLE - 8. Total Extractable Organic Residues (TEO) and Aromatic Hydrocarbon Analytical Blank Values for Tenax Resins (μg), PE Extraction.[a]

Type and Lot No.	Pretreatment[c]	TEO	NAP	2-MN	2,6-DMN	BIPH
Tenax-GC 760511-10	1	8979				
	1	2246				
	2	115				
	2	144				
Tenax-GC 04899-MO3	1	444				
	3	<43	<0.03	<0.03	<0.03	<0.03
	4	29	<0.2	<0.2	<0.2	<0.2
Tenax-TA 04915-A15	1	126	1.6	0.55	0.28	0.38
	2	31	<0.2	<0.2	<0.2	<0.2

---

a) Residues per 10 g Tenax, soxhlet extracted in paper thimbles with PE.

b) TEO eluting between 5-23 minutes and quantified by GC-FID.

c) Pretreatments:
1. None (extracted directly from the bottle)
2. Soxhlet extracted 24 h with PE, vacuum-dried.
3. As in 2, but stored 15 d before re-extraction.
4. As in 2, but stored 120 d before re-extraction.

TABLE 9. Organochlorine Analytical Blank Values for Tenax Resins (ng), PE Extraction.[a]

Type and Lot No.	Pretreatment[b]	p-DCB	o-DCB	1,2,4-TCB	HCB
Tenax-GC 04899-MO3	3	<3	<2	<0.3	<0.3
	4	<16	<11	<1.4	<0.3
Tenax-TA 04915-A15	2	<16	<11	<1.4	<0.3

---

a) Residues per 10 g Tenax, soxhlet extracted with PE in paper thimbles. Extracts cleaned up with 7% fuming H_2SO_4.

b) Pretreatments:
2. Soxhlet extracted 24 h with PE, vacuum-dried.
3. As in 2, but stored 15 d before re-extraction.
4. As in 2, but stored 120 d before re-extraction.

TABLE 10. Organochlorine and Chlorophenol Analytical Blank Values for PUF-Tenax Cartridges (ng), 15% EE-PE Extraction.[a]

Organochlorines	Blank 1[b]	Blank 2[c]
1,2-dichlorobenzene	120	127
1,3-dichlorobenzene		46
1,4-dichlorobenzene	19	<90
1,2,4-trichlorobenzene	1.7	370
1,2,4,5-tetrachlorobenzene		<5
pentachlorobenzene	0.5	<0.5
hexachlorobenzene	0.4	<0.6
heptachlor	<0.5	
Aroclor 1242	53	
Aroclor 1254	26	
α-hexachlorocyclohexane	0.6	<0.7
γ-hexachlorocyclohexane	1.7	<0.8
trans-chlordane	2.8	
cis-chlordane	1.4	
trans-nonachlor	1.0	
Chlorophenols		
2,6-dichlorophenol	<1	<2
2,4-dichlorophenol	<1	<2
3,4-dichlorophenol	<1	<3
2,4,6-trichlorophenol	1.0	1.0
2,3,4,5-tetrachlorophenol	<1	0.3
pentachlorophenol	3.5	0.6

---

a) 10 g Tenax between two 2.5-cm PUF slices, soxhlet extracted with 15% EE-PE. Organochlorine fraction treated with sulfuric acid before analysis. b) Transported to and from the field before analysis. c) Frozen 4 months before analysis.

TABLE 11. Organochlorine and Chlorophenol Analytical Blank Values for PUF-XAD-2 Cartridges (ng), 15% EE-PE Extraction.[a]

	Blank 1	Blank 2	Blank 3	Blank 4[b]
1,3-dichlorobenzene		<88	<75	65
1,4-dichlorobenzene		487	440	<31
1,2-dichlorobenzene	253	295	247	<16
1,2,4-trichlorobenzene	<3.7	92	113	29
1,2,4,5-tetrachlorobenzene	<3.0	<15	<13	<1.0
pentachlorobenzene	<0.3	<2.1	<1.8	<0.2
hexachlorobenzene	0.3	<1.2	<1.1	<0.2
α-hexachlorocyclohexane	<0.2	<1.1	<1.0	<0.1
γ-hexachlorocyclohexane	<0.3	<1.6	<1.4	<0.2
2,6-dichlorophenol	7.6			238
2,4-dichlorophenol	<0.6			7.5
3,4-dichlorophenol	1.1			<2.0
2,4,6-trichlorophenol	0.9			11
2,3,4,5-tetrachlorophenol	0.2			2.2
pentachlorophenol	0.2			8.6

---

a) 14-16 g XAD-2 between two 2.5-cm PUF slices, precleaned by soxhlet extraction with 15% EE-PE (after initial cleanup as in Experimental section), dried, and re-extracted with 15% EE-PE. Chlorobenzene fraction treated with concentrated sulfuric acid before GC-ECD analysis.

b) stored 40 days before re-extraction.

TABLE 12. TEO and Aromatic Hydrocarbon Operational Blank Values for Tenax and PUF-Tenax (μg), PE Extraction.

Trap Type	m^3 air	TEO	NAP	2-MN	2,6-DMN	BIPH
Stacked sieves 3.9 g Tenax/trap	100					
1. (front)		94	0.25	0.26	0.19	2.0
2.		17	<0.03	<0.03	<0.03	<0.03
3.		13	<0.03	<0.03	<0.03	<0.03
4. (back)		17	<0.03	<0.03	<0.03	<0.03
Double sandwich	32					
1. 2 cm PUF		ND[a]	<0.03	<0.03	<0.03	<0.03
2. 10 g Tenax		85	<0.03	<0.03	<0.03	<0.03
3. 2 cm PUF		ND	<0.03	<0.03	<0.03	<0.03
4. 10 g Tenax		17	<0.03	<0.03	<0.03	<0.03
5. 2 cm PUF		ND	ND	ND	ND	ND
Separate front/back cartridges	98					
1. PUF-Tenax (10g Tx)		48	<0.03	<0.03	<0.03	<0.03
2. Tenax (3g)		148	<0.03	<0.03	0.02	0.39
Average blank used in collection experiments with volumes ≤165 m^3 and PE extraction			<0.1	<0.1	<0.1	0.3

---

a) ND = not determined.

TABLE 13. Organochlorine Operational Blank Values for Tenax and PUF-Tenax (ng), PE Extraction.

Trap Type	m^3 air	1,4-DCB	1,2-DCB	1,2,4-TCB	HCB
Double sandwich	34				
1. 2 cm PUF		<2	110	<2	2
2. 10 g Tenax		370	220	20	<1
3. 2 cm PUF		<2	<2	<2	<1
4. 10 g Tenax		<2	<2	<2	<1
5. 2 cm PUF		ND[a]	ND	ND	ND
Double Sandwich	119				
1. 2 cm PUF		2	140	7	6
2. 10 g Tenax		32	<2	<2	<1
3. 2 cm PUF		40	<2	<2	<1
4. 3 g Tenax		90	<2	<2	<1
5. 2 cm PUF		ND	ND	ND	ND
Separate front/back cartridges	98				
1. PUF-Tenax (10g Tx)		<2	80	11	0.2
2. Tenax (3g)		13	18	6	0.6
Average blank used in collection experiments with volumes ≤ 165 m^3 and PE extraction.		56	58	6	1.5

---

a) ND = not determined.

TABLE _14. Organochlorine Operational Blanks for PUF-Tenax (ng), 15% EE-PE Extraction.

	Blank 1, 314 m^3		Blank 2, 288 m^3
	Front	Back	Front
1,2-dichlorobenzene	832	1311	196
1,3-dichlorobenzene	289	345	876
1,4-dichlorobenzene	601	855	1456
1,2,4-trichlorobenzene	190	306	159
1,2,4,5-tetrachlorobenzene	29	63	8.2
pentachlorobenzene	2.5	8.2	1.8
hexachlorobenzene	1.4	3.7	2.1
α-hexachlorocyclohexane	4.5	12.8	4.5
γ-hexachlorocyclohexane	2.5	4.2	1.4

TABLE 15. Organochlorine and Chlorophenol Operational Blanks for PUF-XAD-2 (ng), 15% EE-PE Extraction.

	Blank 1 368 m^3	Blank 2 354 m^3		Blank 3 361 m^3	
	Front	Front	Back	Front	Back
Chlorobenzenes					
1,2-dichlorobenzene	239	487	202	<70	<70
1,3-dichlorobenzene	258	139	75	<53	<53
1,4-dichlorobenzene	562	751	178	<126	<126
1,2,4-trichlorobenzene	96	190	156	268	438
1,2,4,5-tetrachlorobz.	16	<9	81	<15	<13
pentachlorobenzene	1	2	<0.7	5	10
hexachlorobenzene	1	1	1	3	6
α-hexachlorocyclohexane	3	2	1	6	14
γ-hexachlorocyclohexane	1	1	<0.6	3	8
Chlorophenols					
2,6-dichlorophenol	101			7.1	7.3
2,4-dichlorophenol	46			22	21
3,4-dichlorophenol	<3			6.3	10
2,4,6-trichlorophenol	11			2.5	3.2
2,3,4,6-tetrachlorophenol	7.1			2.2	6.3
pentachlorophenol	3.1			5.8	13

TABLE 16. SOC Quantities Used in Collection Experiments, µg.

	High Level Experiments	Low Level Experiments
Aromatic Hydrocarbons		
naphthalene	259-752	30
2-methylnaphthalene	152-440	17
2,6-dimethylnaphthalene	204-593	23
biphenyl	175-508	20
Organochlorines		
1,2-dichlorobenzene	15-43	1.5-3.0
1,3-dichlorobenzene		2.0-4.1
1,4-dichlorobenzene	15-21	2.1-4.3
1,2,4-trichlorobenzene	2-15	0.32-0.64
1,2,4,5-tetrachlorobenzene		0.20-0.40
pentachlorobenzene		0.021-0.043
hexachlorobenzene	0.1-0.6	0.013-0.026
α-hexachlorocyclohexane		0.013-0.030
γ-hexachlorocyclohexane		0.017-0.035
Chlorophenols		
2,4-dichlorophenol		0.35-0.96
2,6-dichlorophenol		0.37-1.0
3,4-dichlorophenol		0.70-2.0
2,4,6-trichlorophenol		0.17-0.46
2,3,4,5-tetrachlorophenol		0.077-0.21
pentachlorophenol		0.043-0.12

TABLE 17. Average Operational Blanks (ng) Used for Correcting Recoveries of PUF-Tenax and PUF-XAD-2 Experiments (15% EE-PE Extraction).

	PUF-Tenax[a]	PUF-XAD-2[b]
1,2-dichlorobenzene	780 ± 559	213 ± 171
1,3-dichlorobenzene	503 ± 324	116 ± 87
1,4-dichlorobenzene	971 ± 439	348 ± 290
1,2,4-trichlorobenzene	218 ± 77	230 ± 132
1,2,4,5-tetrachlorobenzene	27 ± 23	12 ± 21
pentachlorobenzene	4.4 ± 2.5	3.2 ± 3.2
hexachlorobenzene	2.3 ± 0.9	2.4 ± 2.2
α-hexachlorocyclohexane	6.2 ± 2.0[c]	5.2 ± 5.2
γ-hexachlorocyclohexane	2.9 ± 1.1	2.7 ± 3.1

---

a) Table 14 values for DCBs and TCB, Table 14 plus back trap values from CE 23 and 27 (Appendix) for TeCB, PeCB, HCB, and HCHs.

b) Table 15 values.

c) Excludes one blank of 12.8 ng.

TABLE 18. Aromatic Hydrocarbon Collection on Tenax and PUF-Tenax, High Level Experiments.

	% Recovery[a]			
Trap Type and m^3 Air	NAP	2-MN	2,6-DMN	BIPH
Stacked Sieves,[b] each containing 3.9 g Tenax				
100	92	97	95	93
Double Sandwich Cartridges[c] Tenax = 10 g front, 3 g back				
34	96	97	98	98
34	79	82	83	85
117	.175[d]	92	98	97
119	91	93	95	94
165	79	81	81	80

---

a) Based on sum of quantities found in multiple traps.

b) Figure 4.

c) Figure 5.

d) Possible contamination from use of XAD-2 in prefilter.

TABLE 19. Aromatic Hydrocarbon Collection on Tenax and PUF-Tenax, Low-Level Experiments

	% Recovery[a]			
Trap type and m^3	NAP	2-MN	2,6-DMN	BIPH
Double sandwich[b] cartridges, Tenax = 10 g front, 3 g back				
78	88	81	78	78
Separate cartridges[c] Tenax = 10 g front, 3 g back				
102	74	77	88	85
125	76	85	82	80

---

a) Based on sum of quantities found in multiple traps.

b) Figure 5.

c) Figure 6.

TABLE 20. Chlorobenzene Collection on Tenax and PUF-Tenax, High Level Experiments.

	% Recovery[a]			
Trap Type and m^3 Air	1,2-DCB	1,4-DCB	1,2,4-TCB	HCB
Stacked Sieves,[b] each containing 3.9 g Tenax				
100	55		67	93
Double Sandwich Cartridges[c] Tenax = 10 g front, 3 g back				
32	88	87	107	77
34	76	85	108	125
117	77	74	95	98
165	86	76	113	101

---

a) Based on sum of quantities found in multiple traps.

b) Figure 4.

c) Figure 5.

TABLE 21 . Organochlorines Collection on PUF-Tenax, Low-Level Experiments.

	% Recovery[a]								
Trap Type and m^3	o-DCB	m-DCB	p-DCB	1,2,4-TCB	1,2,4,5-TeCB	PeCB	HCB	α-HCH	γ-HCH
Double Sandwich Cartridges[b], Tenax = 10g front, 3 g back.									
78	91		54	111			106		
Separate cartridges[c], Tenax = 10 g front, 3 g back									
102	80		77	95			81		
125	71		65	90			93		
346					89	93	98	68	112
Separate cartridges[d], Tenax = 5 g front, 5 g back									
285					87	87	76	79	85
334					70	55	48	29	39
353					88	99	94	66	86
358					104	102	109	79	83

a) Based on sum of quantities found in multiple traps. b) Figure 5

c) Figure 6 d) Figure 7

TABLE 22. Collection of Organochlorines on PUF-XAD-2, Low-Level Experiments.

	% Recovery[a]				
Trap type and m^3	1,2,4,5-TeCB	PeCB	HCB	α-HCH	γ-HCH
Separate cartridges[b] XAD = 14-16 g front, 14-16 g back					
348	104	80	78	58	58
367	78	65	51	38	49
372	110	91	72	56	53
372	132	167	132	102	109

a) Based on sum of quantities found in multiple traps.

b) Figure 7.

TABLE 23. Organochlorines Collection on PUF-Florisil, Low-Level Experiments.

Trap Type and m^3	% Recovery[a]								
	o-DCB	m-DCB	p-DCB	TCB	TeCB	PeCB	HCB	α-HCH	γ-HCH
Separate cartridges[b] Florisil = 30 g front, 30 g back									
359	<1	<2	<1	34	58	89	77	66	51
371	46	7	7	88	64	82	103	89	76
371	4	<1	<2	36	69	71	79	139	65

a) Based on sum of quantities in multiple traps.

b) Figure 7.

TABLE 24. Collection of Chlorophenols on PUF-Tenax

	% Recovery[a]					
Trap type and m^3	2,4-DCP	2,6-DCP	3,4-DCP	2,4,6-TCP	2,3,4,5-TeCP	PCP
Separate cartridges[b] Tenax = 10 g front, 3 g back						
346	149	106	116	106	65	65
Separate cartridges[c] Tenax = 5 g front, 5 g back						
285	53	48	55	25	29	10
288[d]	84	87	86	73	74	
314	63	58	31	27	18	11
334	112	109	111	89	97	39
353	118	105	100	95	95	39
358	109	94	85	90	66	

TABLE 24. Collection of Chlorophenols on PUF-Tenax

a) Based on sum of quantities found in multiple traps.

b) Figure 6

c) Figure 7

d) Front trap = 6.7 g Tenax, compounds spiked onto a filter directly in front of adsorbent cartridge instead of being injected into mixing chamber.

TABLE 25. Chlorophenol Collection on PUF-XAD-2.

Trap type and m^3	% Recovery[a]					
	2,4-DCP	2,6-DCP	3,4-DCP	2,4,6-TCP	2,3,4,5-TeCP	PCP
Separate cartridges[b] XAD = 14-16g front, 14-16 g back						
348	17	56	25	35	23	5
354[c]	79	71	96	81	109	37
367	79	67	65	26	26	10
372	121	110	88	97	70	29
372	90	79	75	69	35	26

a) Based on sum of quantities found in multiple traps.

b) Figure 7.

c) Compounds spiked directly onto front trap instead of being injected into mixing chamber.

TABLE 26. Chlorophenols Collection on PUF-Florisil.

Trap Type and m^3	% Recovery[a]					
	2,4-DCP	2,6-DCP	3,4-DCP	2,4,6-TCP	2,3,4,5-TeCP	PCP
Separate cartridges[b] Florisil = 30 g front, 30 g back						
359	41	61	66	51	36	16
366	10	51	76	63	60	67
371	17	27	12	16	8	6

a) Based on sum of quantities found in multiple traps.

b) Figure 7.

TABLE 27. Breakthrough of Aromatic Hydrocarbons and Dichlorobenzenes from Front to Back PUF-Tenax Adsorbent Traps[a].

	Percent on Back Traps[b]		
Air volume, m^3	NAP	1,2-DCB	1,4-DCB
32		4	4
34	<1	1	3
34	2		
78	4	6	1
102	3	6	6
117	8	10	6
119	<1		
125	<2	14	8
165	3	11	11

---

a) Double sandwich (Figure 5) or separate front sandwich and back Tenax (Figure 6) traps; Tenax = 10 g front, 3 g back.

b) Percent of total recovered. Breakthrough of heavier 2-ring aromatic hydrocarbons and chlorobenzenes was <2%.

TABLE 28. Average Recoveries of Heavier Chlorobenzenes and Hexachlorocyclohexanes in 285-372 m^3 Air Collection Experiments.

	Recovery: Range and (Mean ± S.D.)		
	PUF-Tenax[a]	PUF-XAD-2[b]	PUF-Florisil[c]
1,2,4,5-TeCB	87-104 (92±8)	78-132 (106±22)	58-69 (63±6)
PeCB	87-102 (95±7)	65-91 (79±13)	71-89 (81±9)
HCB	76-109 (94±14)	51-132 (83±35)	77-103 (86±14)
α-HCH	66-79 (73±7)	38-102 (64±27)	66-139 (98±37)
γ-HCH	83-112 (92±14)	49-109 (67±28)	51-76 (64±13)

a) n = 4 (one out of five experiments excluded).

b) n = 4, one experiment excluded for PeCB (n = 3).

c) n = 3.

FIGURE 1. General Metal Works PS-1 hi-vol air sampler.

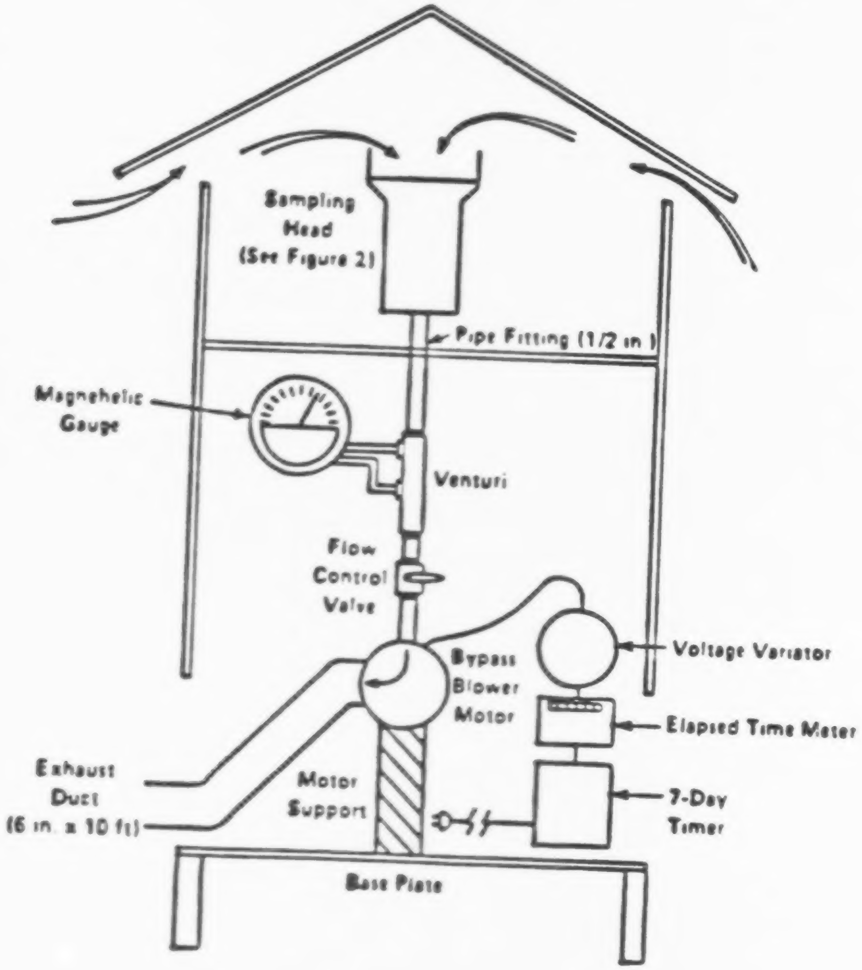

Sampling
Head
(See Figure 2)
Pipe Fitting (1/2 in.)
Magnehelic
Gauge
Venturi
Flow
Control
Valve
Voltage Variator
Bypass
Blower
Motor
Elapsed Time Meter
Exhaust
Duct
(6 in. x 10 ft)
Motor
Support
7-Day
Timer
Base Plate

FIGURE 2. Exploded view of PS-1 sampling head.

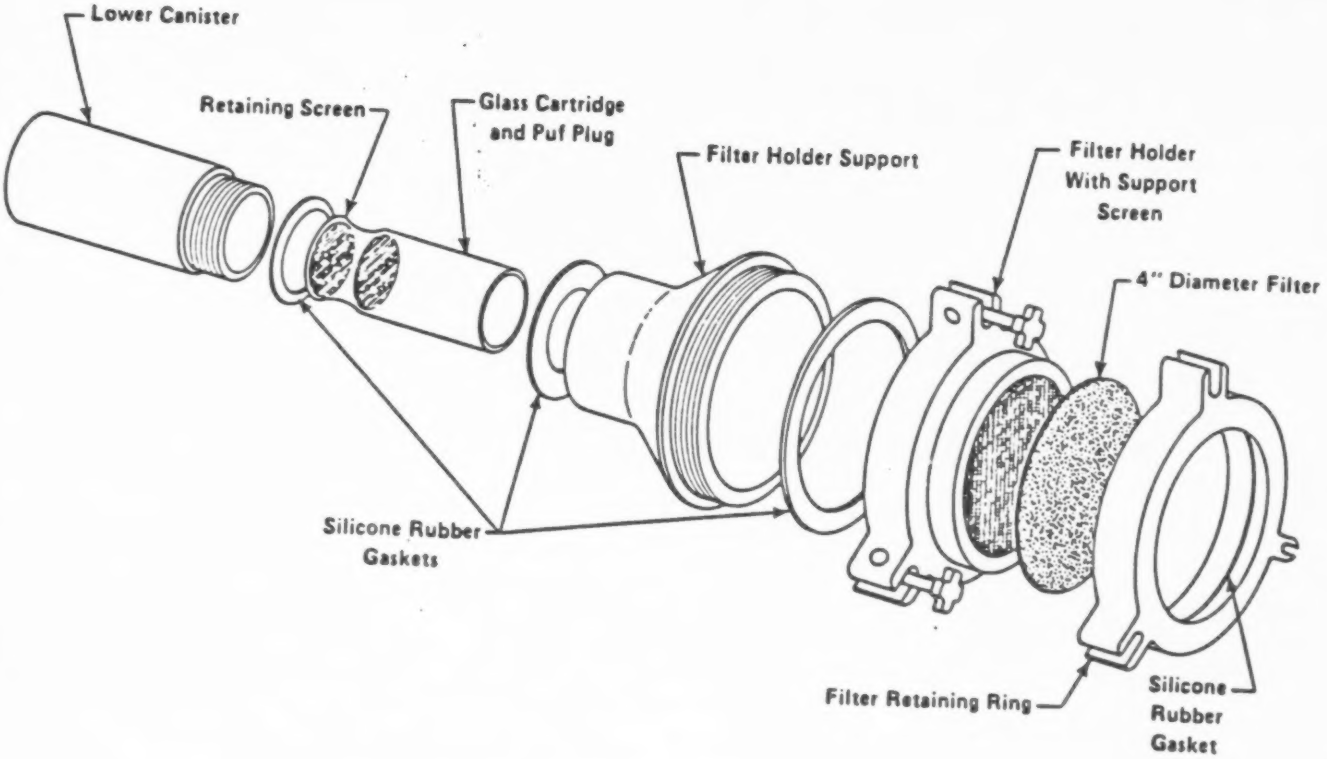

Lower Canister
Retaining Screen
Glass Cartridge and Puf Plug
Filter Holder Support
Filter Holder With Support Screen
4" Diameter Filter
Silicone Rubber Gaskets
Filter Retaining Ring
Silicone Rubber Gasket

FIGURE 3. Sampling train, showing prefilter, sample inlet, mixing chamber, and sandwich adsorbent cartridge.

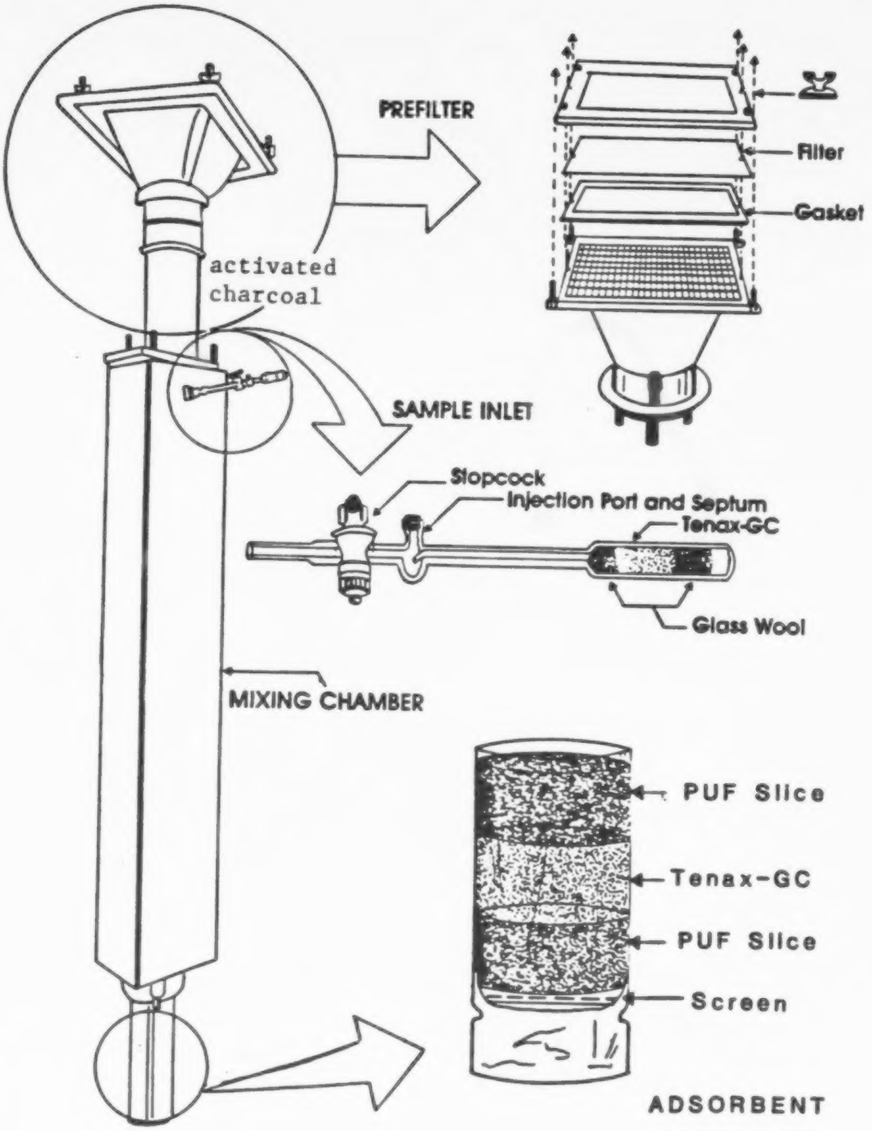

PREFILTER
Filter
Gasket
activated charcoal
SAMPLE INLET
Stopcock
Injection Port and Septum
Tenax-GC
Glass Wool
MIXING CHAMBER
PUF Slice
Tenax-GC
PUF Slice
Screen
ADSORBENT CARTRIDGE

FIGURE 4. Stacked sieve Tenax traps.

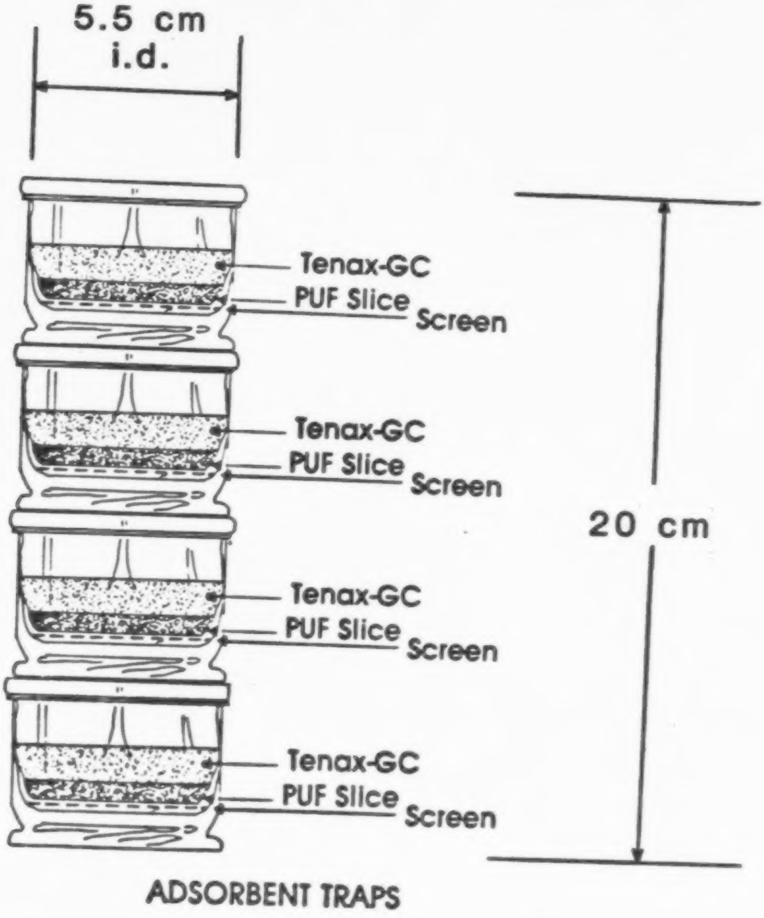

5.5 cm
i.d.
Tenax-GC
PUF Slice
Screen
Tenax-GC
PUF Slice
Screen
Tenax-GC
PUF Slice
Screen
Tenax-GC
PUF Slice
Screen
20 cm
ADSORBENT TRAPS

FIGURE 5. Double sandwich PUF-Tenax cartridge.

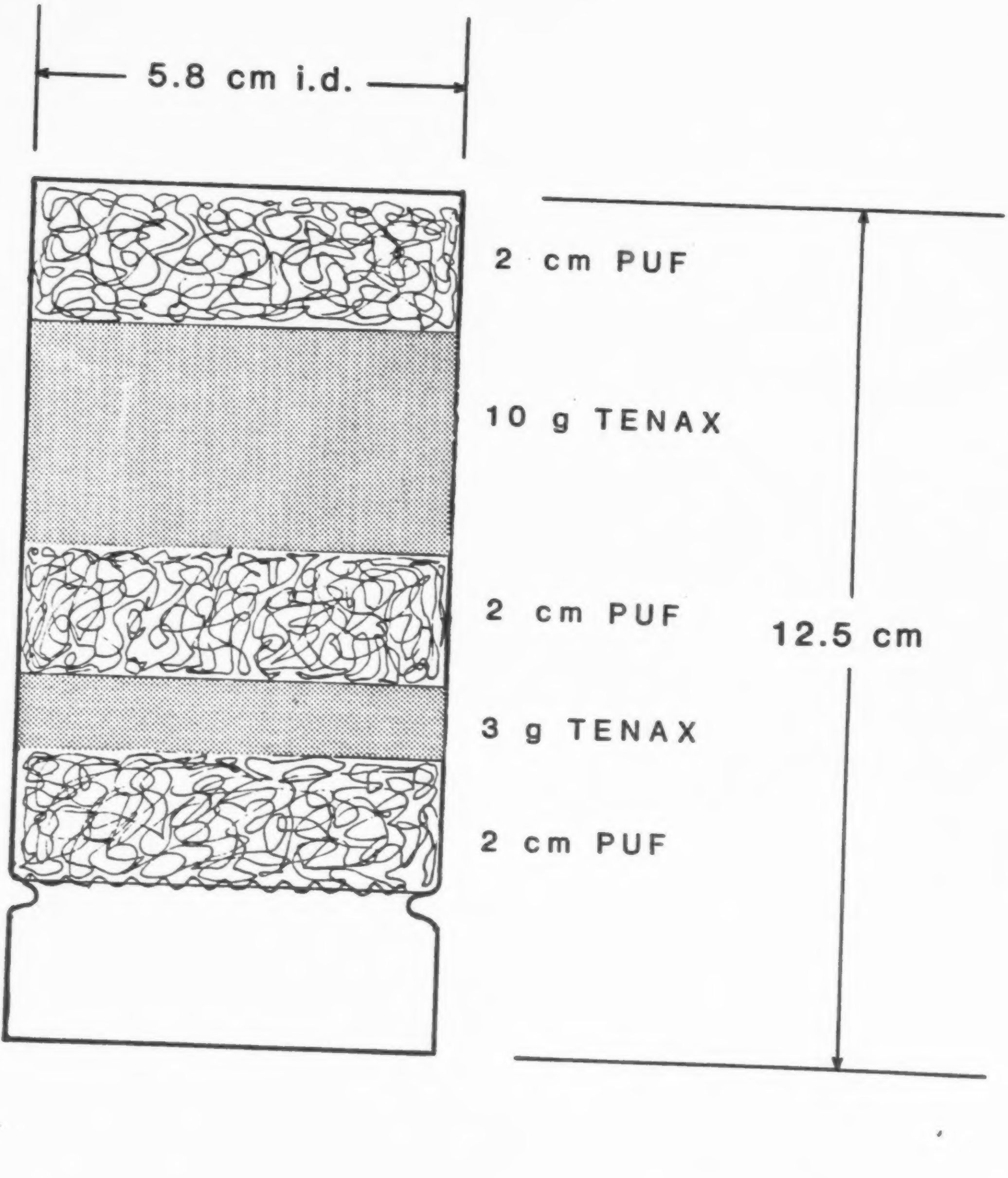

5.8 cm i.d.
2 cm PUF
10 g TENAX
2 cm PUF
12.5 cm
3 g TENAX
2 cm PUF

FIGURE 6. Separate front sandwich and back Tenax traps.

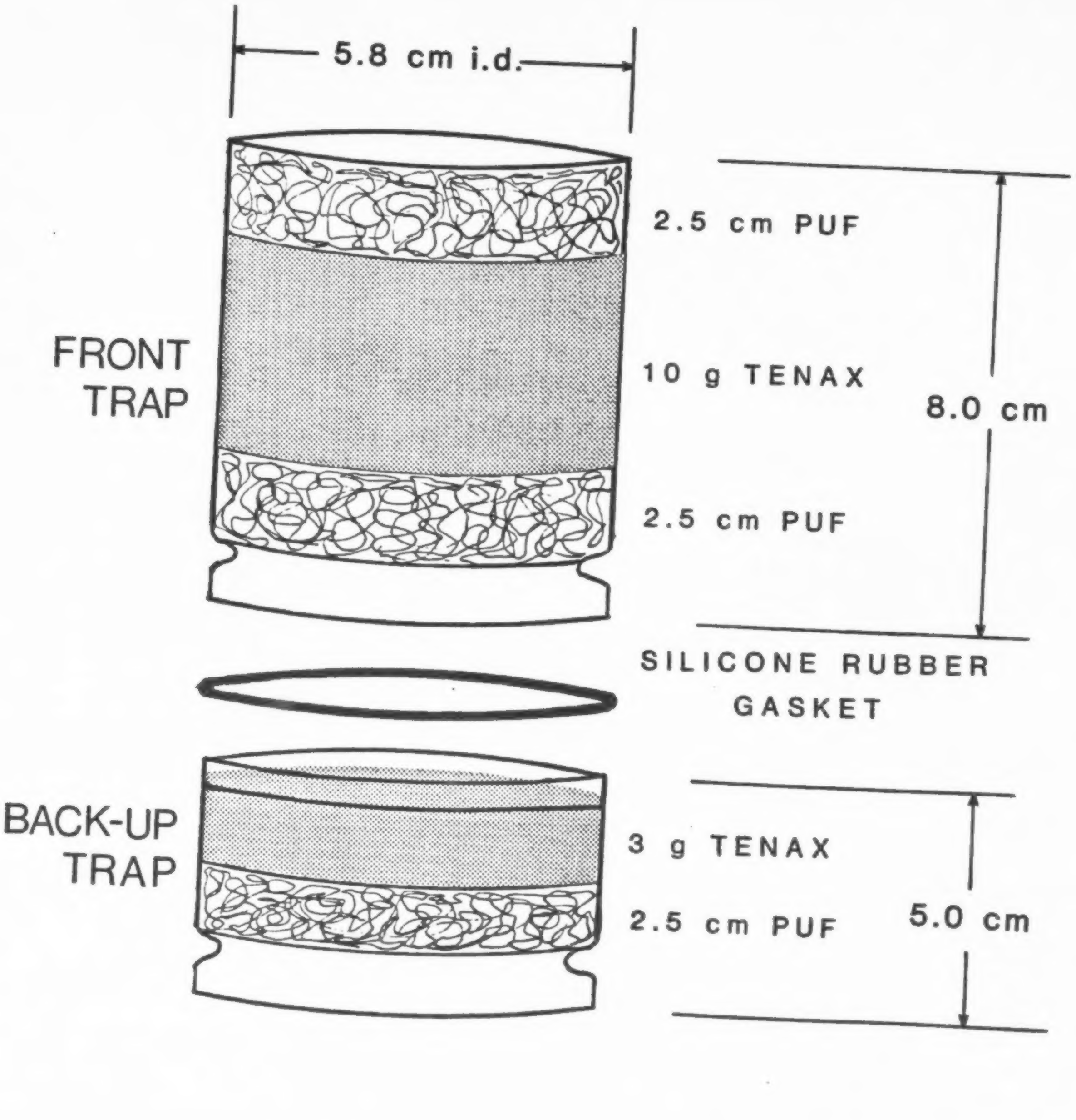

5.8 cm i.d.
2.5 cm PUF
FRONT TRAP
10 g TENAX
8.0 cm
2.5 cm PUF
SILICONE RUBBER GASKET
BACK-UP TRAP
3 g TENAX
2.5 cm PUF
5.0 cm

FIGURE 7. Separate front and back sandwich traps with Tenax. This design was also used with XAD-2 and Florisil (quantities given in text).

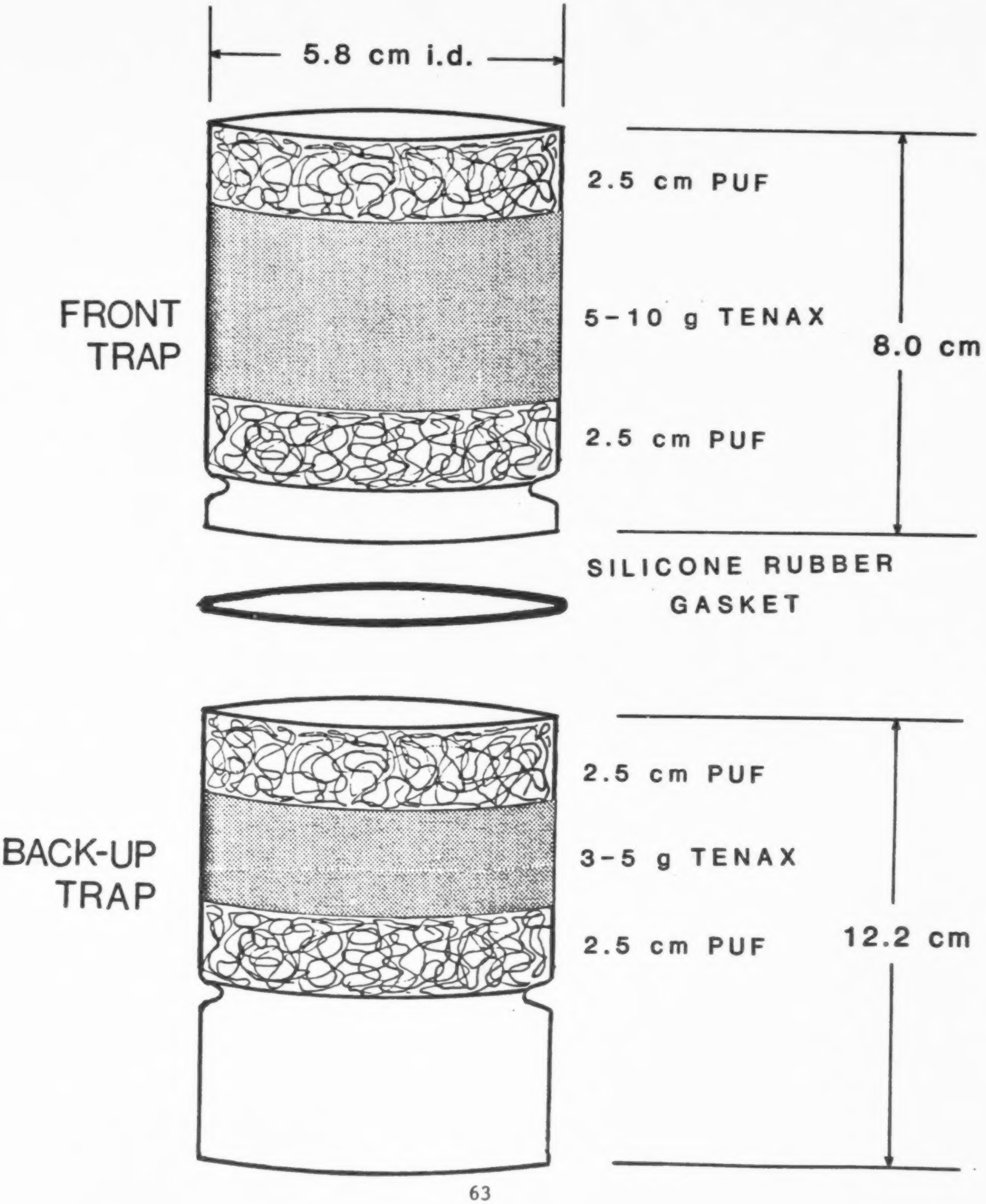

5.8 cm i.d.
2.5 cm PUF
5-10 g TENAX
8.0 cm
FRONT TRAP
2.5 cm PUF
SILICONE RUBBER GASKET
2.5 cm PUF
BACK-UP TRAP
3-5 g TENAX
2.5 cm PUF
12.2 cm

FIGURE 8. Initial extract of 15 g Tenax (middle) and 3.1 g precleaned Tenax (bottom). A standard mixture of n-alkanes is shown at the top. GC-FID.

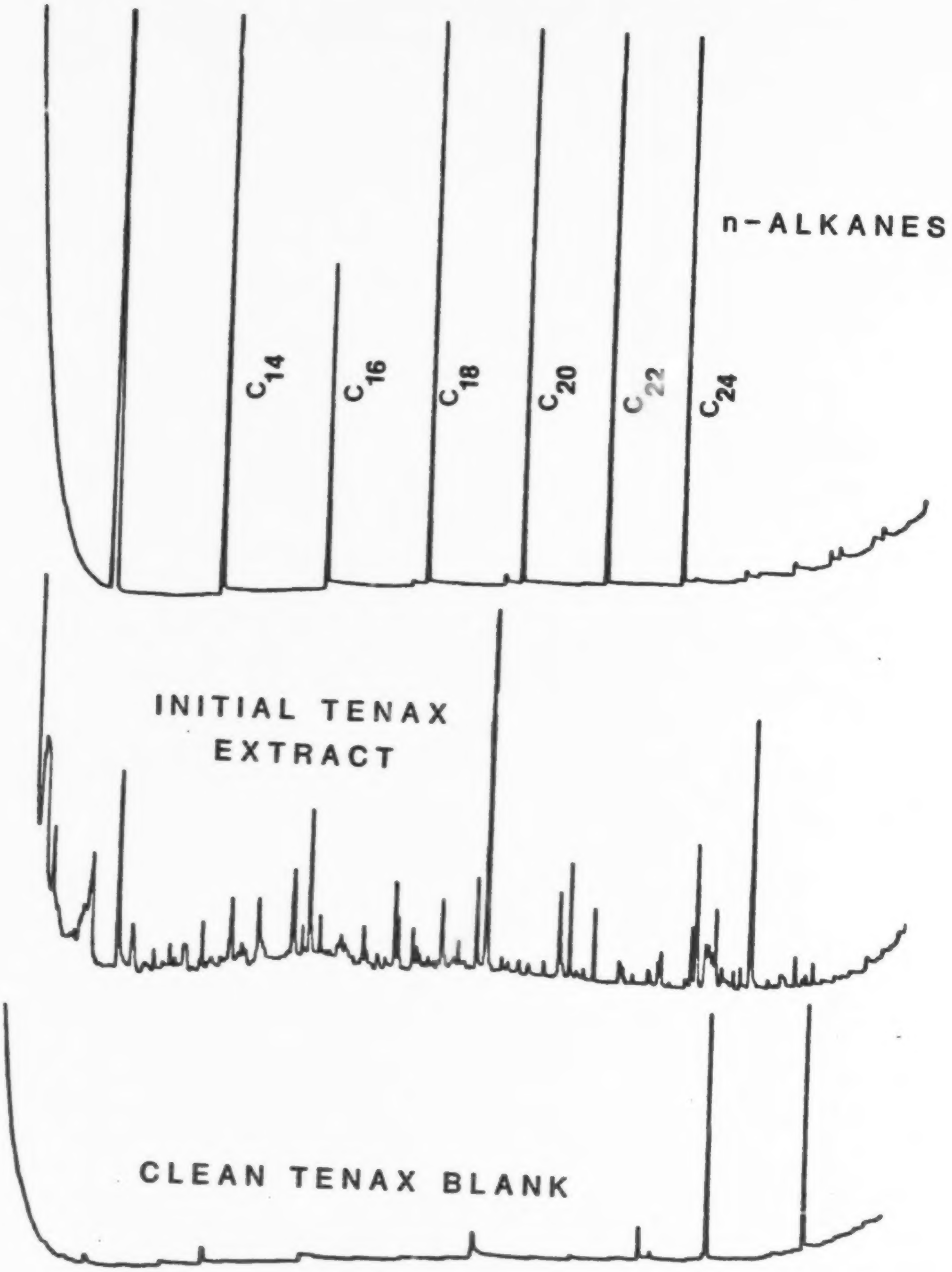


FIGURE 9. Tenax blank (3.6 g), extracted after 4 months' storage A standard mixture of 2-ring aromatic hydrocarbons is shown at the top. GC-FID.

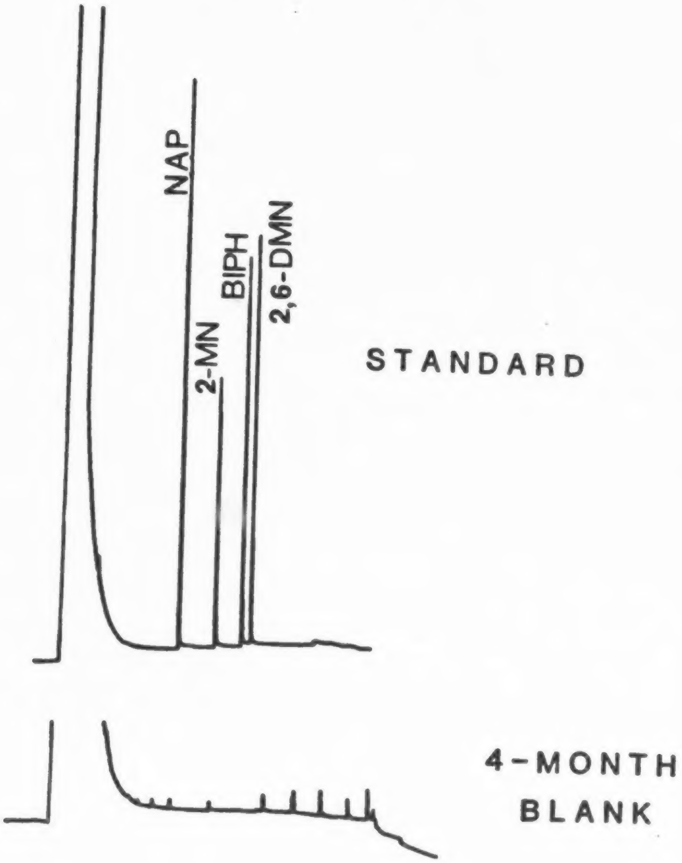


FIGURE 10. Initial extract of 15 g Tenax and a 3.6-g Tenax blank that had been stored for 4 months. GC-ECD after sulfuric acid cleanup.

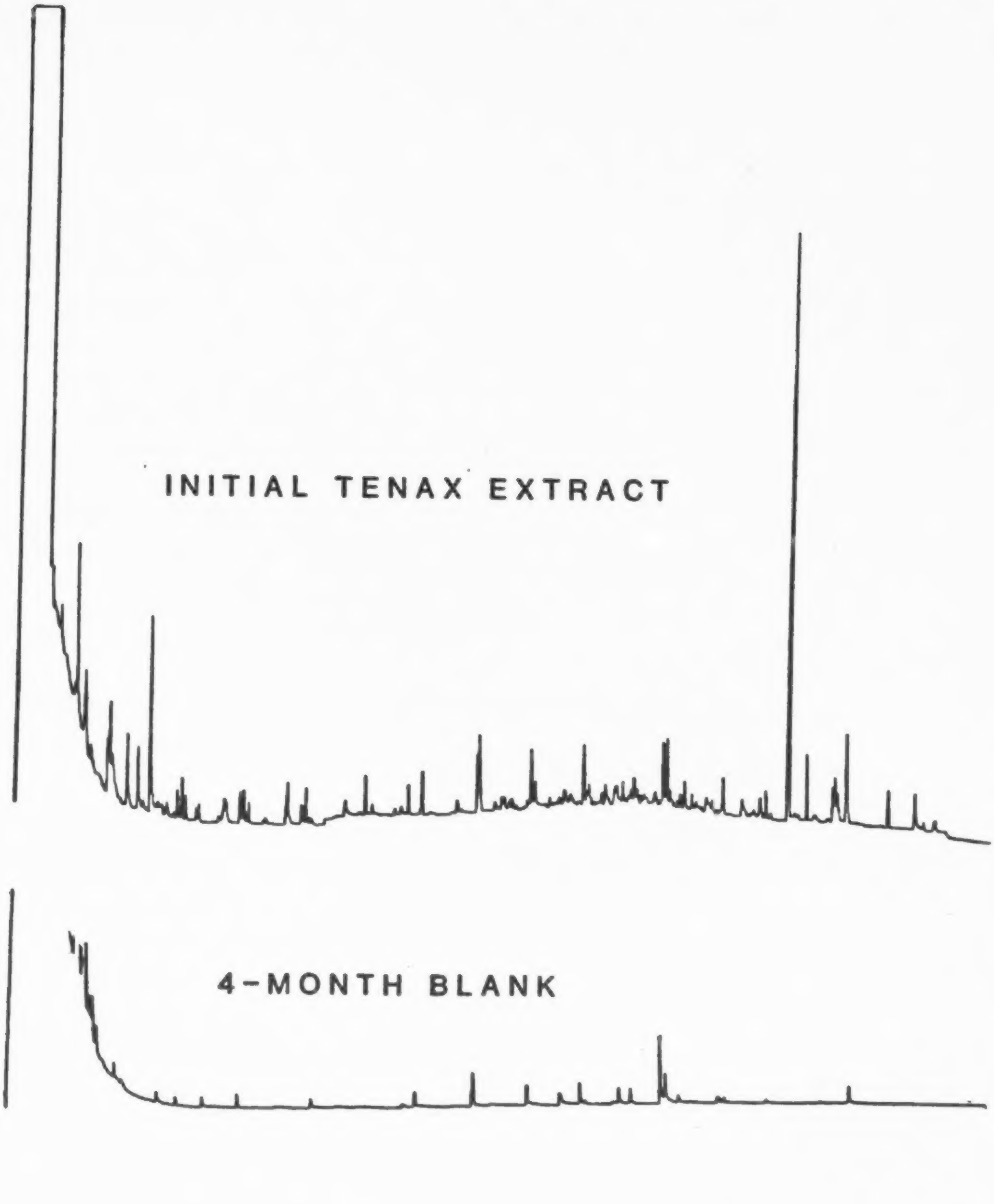


FIGURE 11. PE extracts of a Tenax blank before and after sulfuric acid cleanup. GC-ECD.

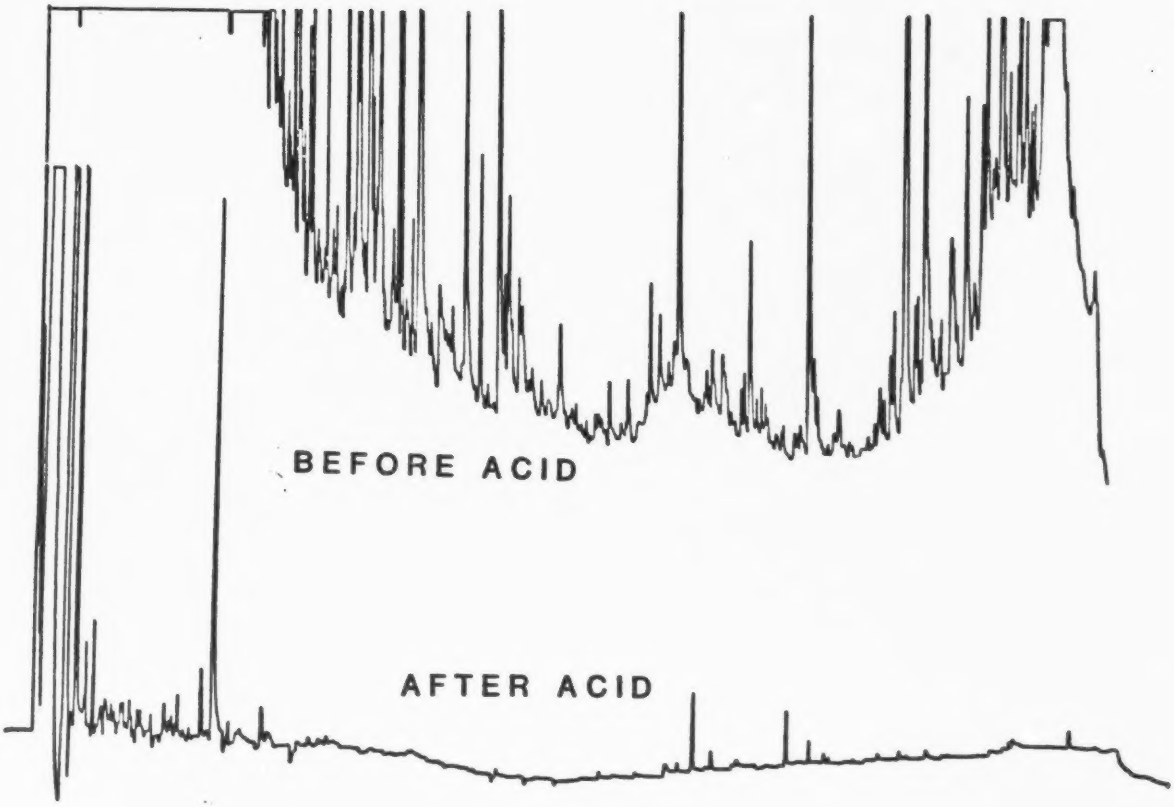


FIGURE 12. As in Fig. 11, but using 15% EE-PE as the extracting solvent. GC-ECD.

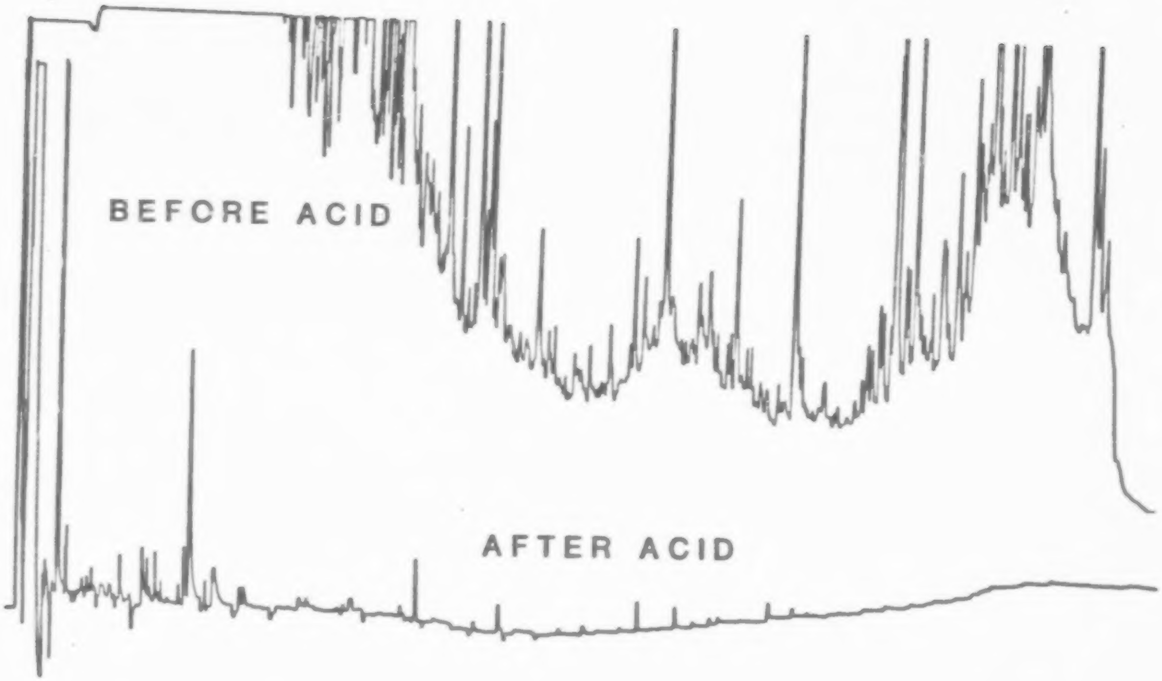


FIGURE 13. Analytical blank: PUF-Tenax trap, cleaned up and stored 4 months (frozen) before analysis. GC-ECD after sulfuric acid cleanup.

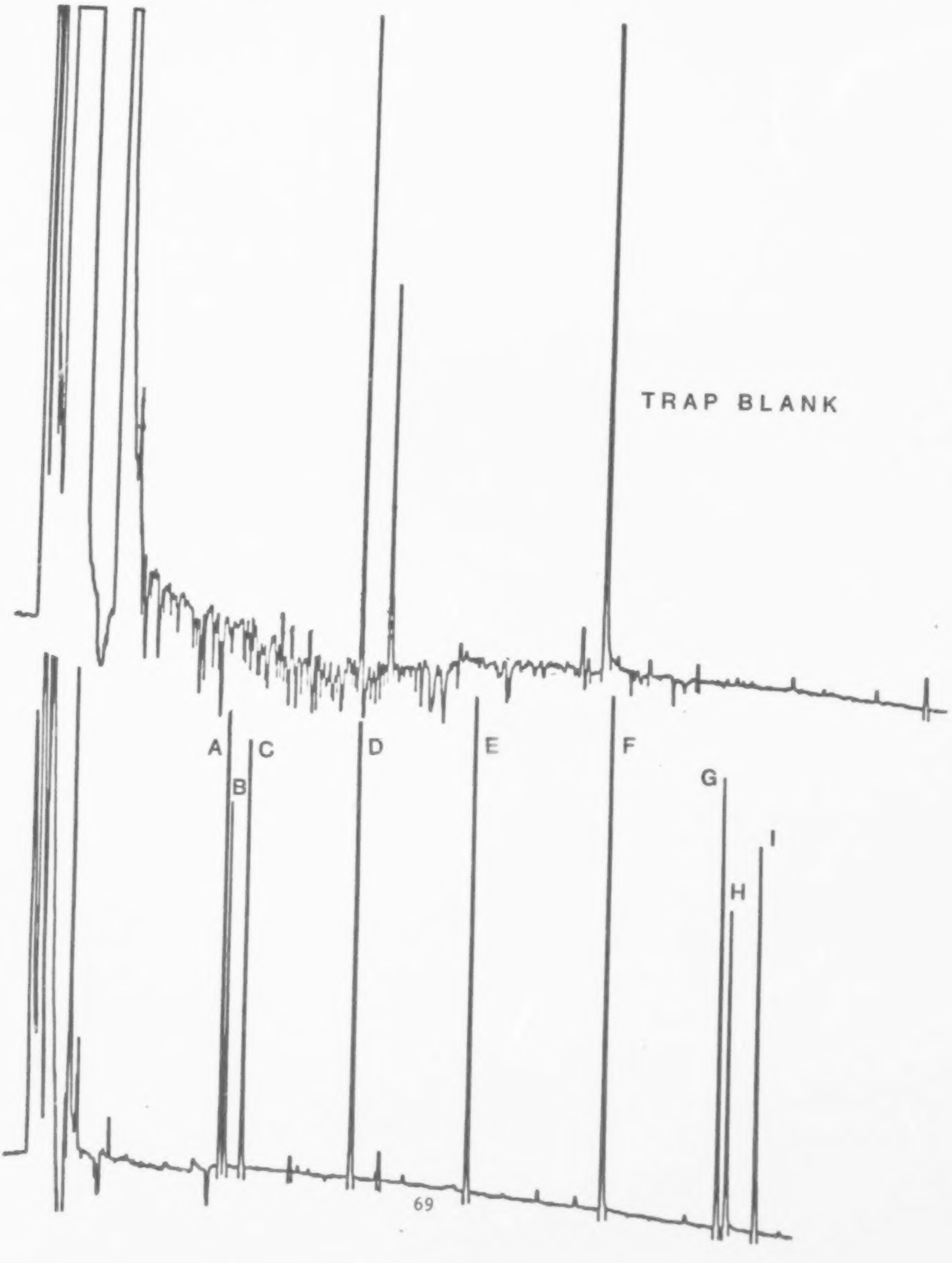


FIGURE 14. PUF-Tenax operational blank, 98 m^3 air, PE extraction. Front and back traps are the types shown in Fig. 6. A standard mixture of chlorobenzenes is shown at the top. GC-ECD after sulfuric acid cleanup.

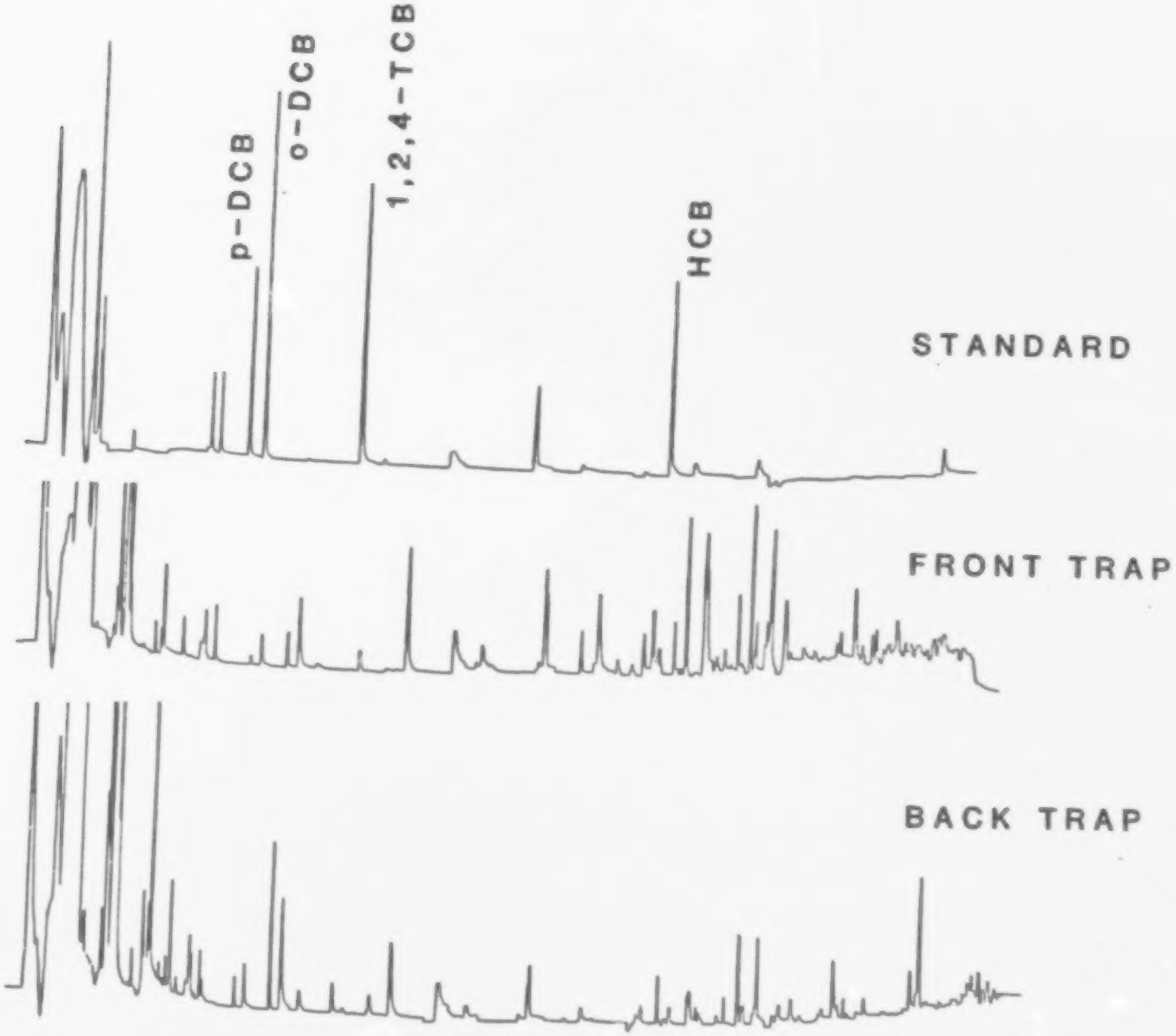


FIGURE 15. PUF-Tenax operational blank, 288 m^3, 15% EE-PE extraction. Trap is the type shown in Figure 7. A standard mixture of organochlorines is shown at the bottom; retention times correspond to the following compounds: 5.36 = m-DCB, 5.49 = p-DCB, 5.98 = o-DCB, 9.20 = 1,2,4-TCB, 12.77 = 1,2,4,5-TeCB, 17.03 = PeCB, 20.71 = α-HCH, 20.94 = HCB, 21.97 = γ-HCH. GC-ECD after sulfuric acid cleanup.

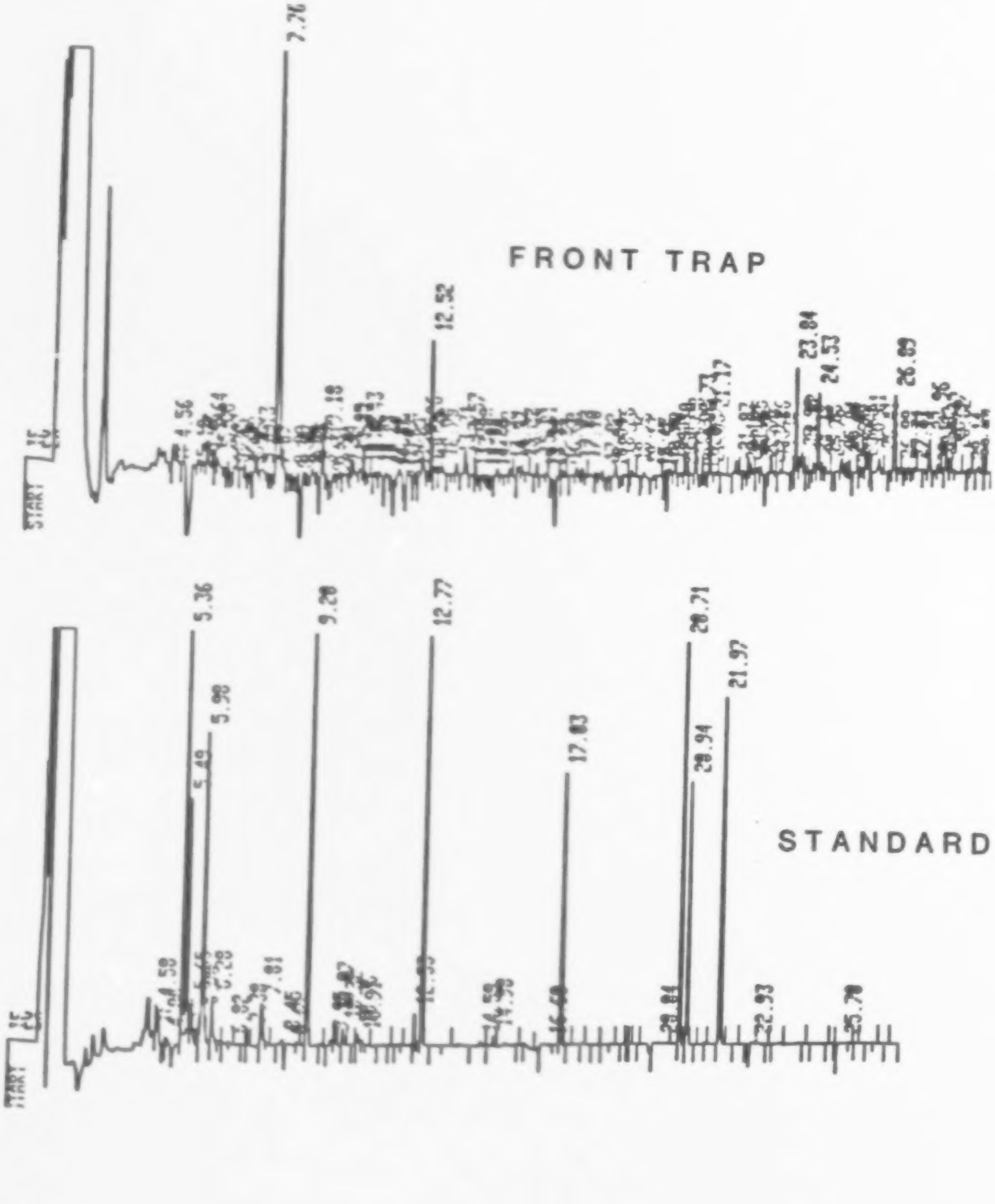


FIGURE 16. PUF-XAD-2 operational blank, 354 m^3 air, 15% EE-PE extraction. Front and back traps are the types shown in Fig. 6. A standard mixture of chlorobenzenes and HCHs is shown at the bottom. A = m-DCB, B = p-DCB, C = o-DCB, D = 1,2,4-TCB, E = 1,2,4,5-TeCB, F = PeCB, G = α-HCH, H = HCB, I = γ-HCH. GC-ECD after sulfuric acid cleanup.

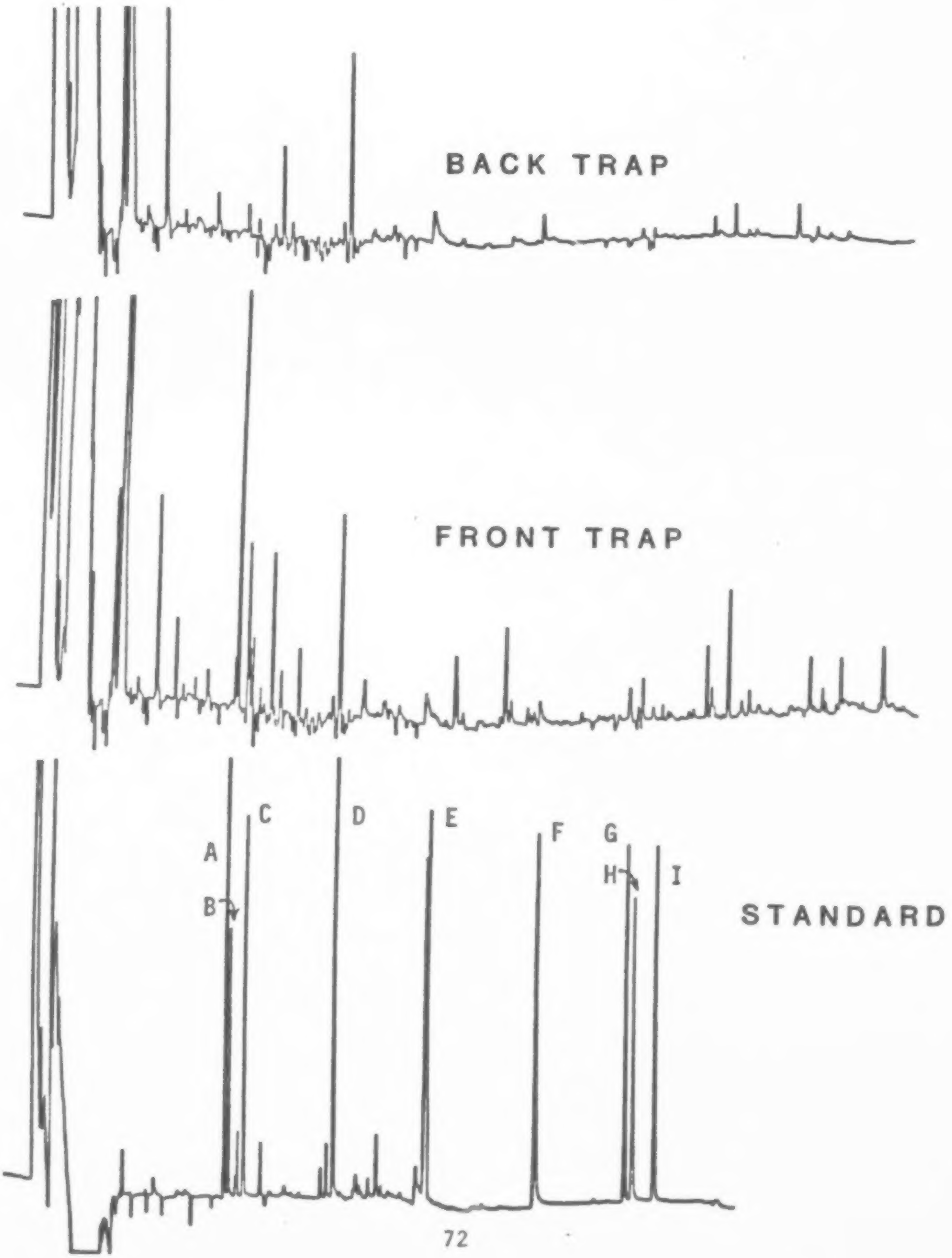


FIGURE 17. Front trap extract of PUF-Tenax collection experiment (Table A14), showing the UCM. GC-ECD after sulfuric acid cleanup.

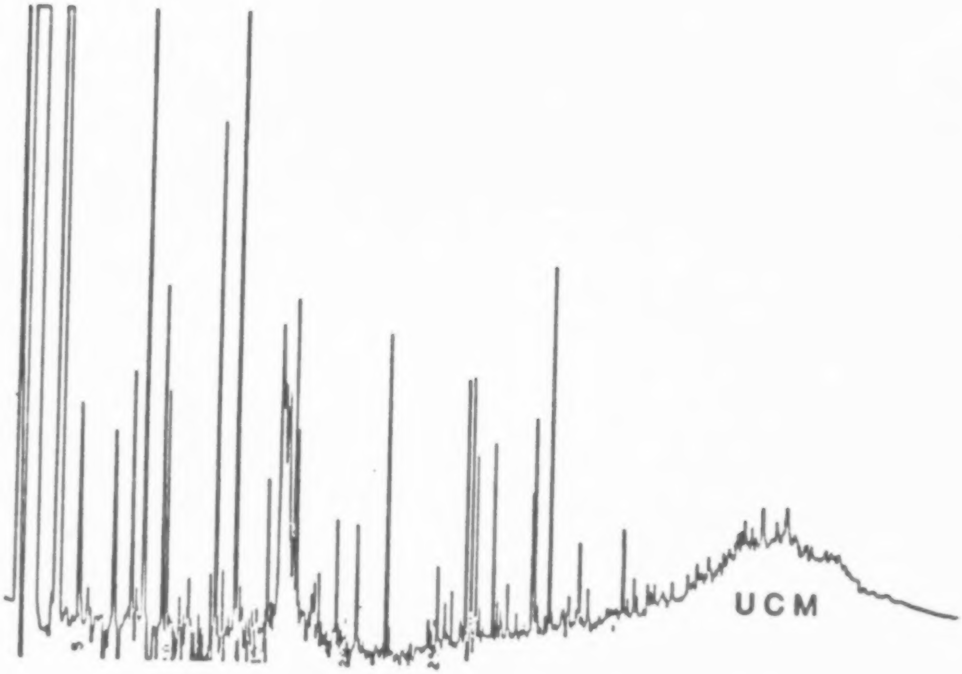


FIGURE 18. Front and back PUF-Tenax trap extracts from December, 12, 1985 ambient air sample, and a blank trap extract. Note the UCM in the front trap chromatogram and lack of the UCM in the back trap chromatogram. GC-ECD after sulfuric acid cleanup.

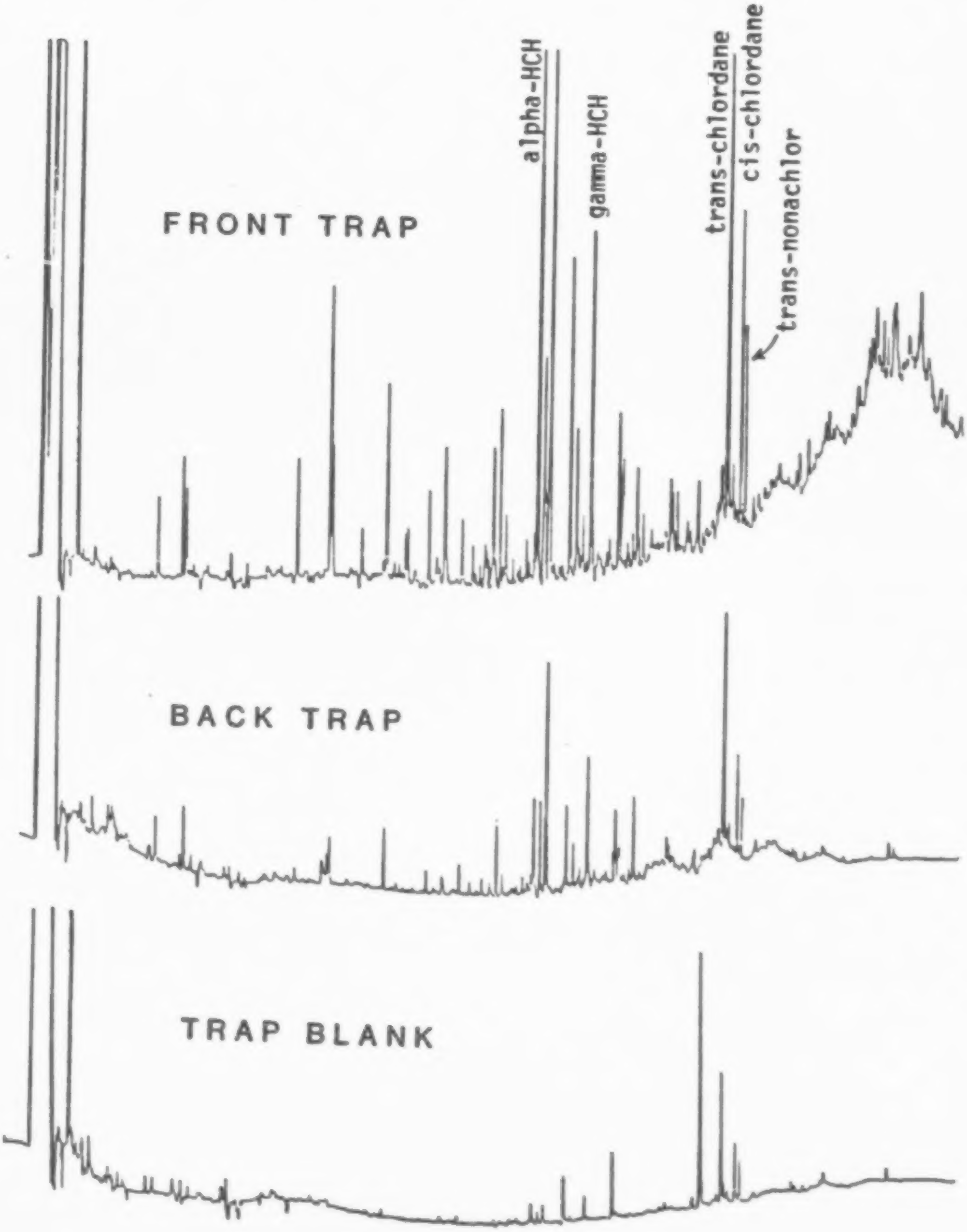


FIGURE 19. UCM section of Fig. 18 expanded. Note that the UCM elutes after most of the toxaphene fingerprint.

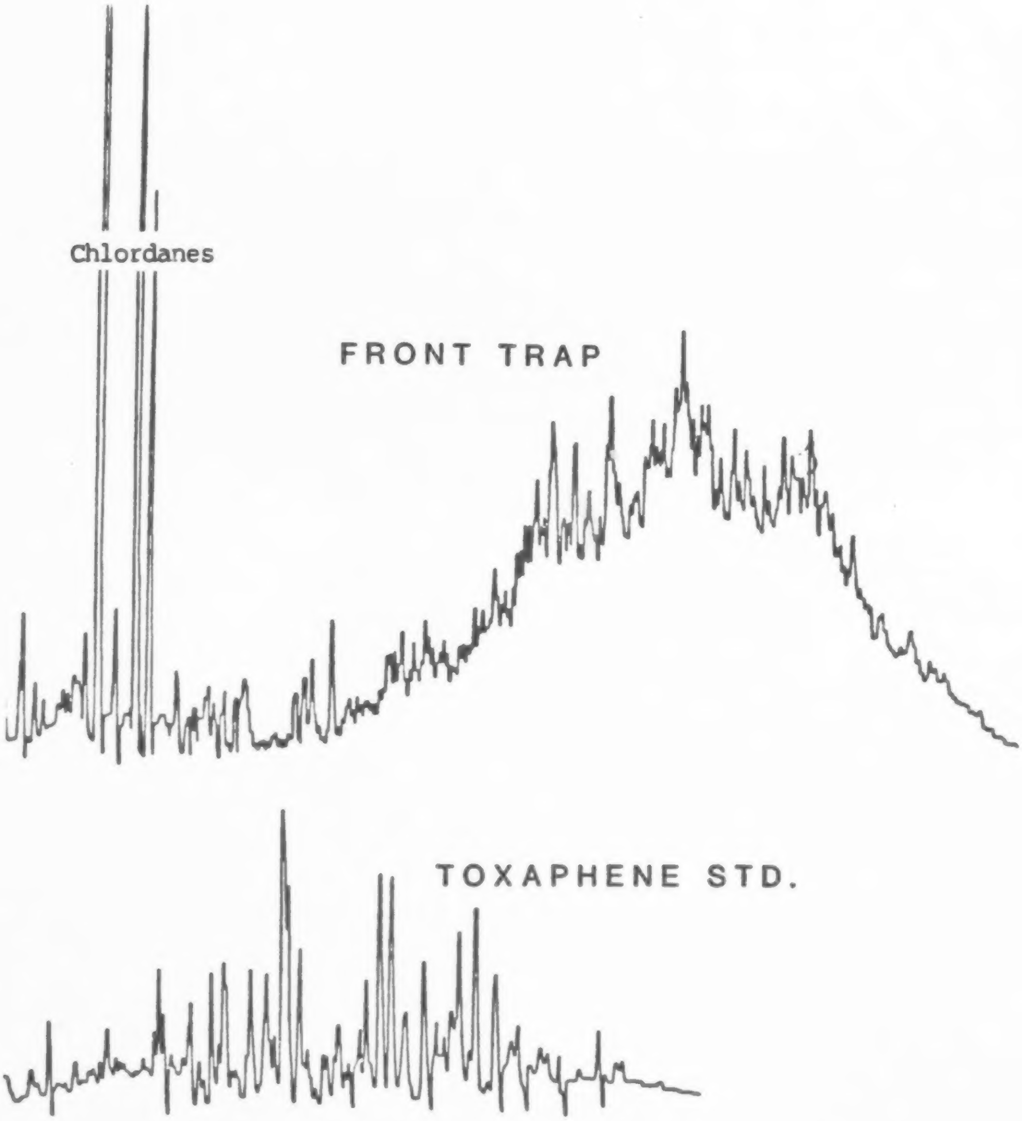


FIGURE 20. Front and back trap extracts from collection experiment 11 , 125 m^3 air (Table A11). GC-ECD after sulfuric acid cleanup.

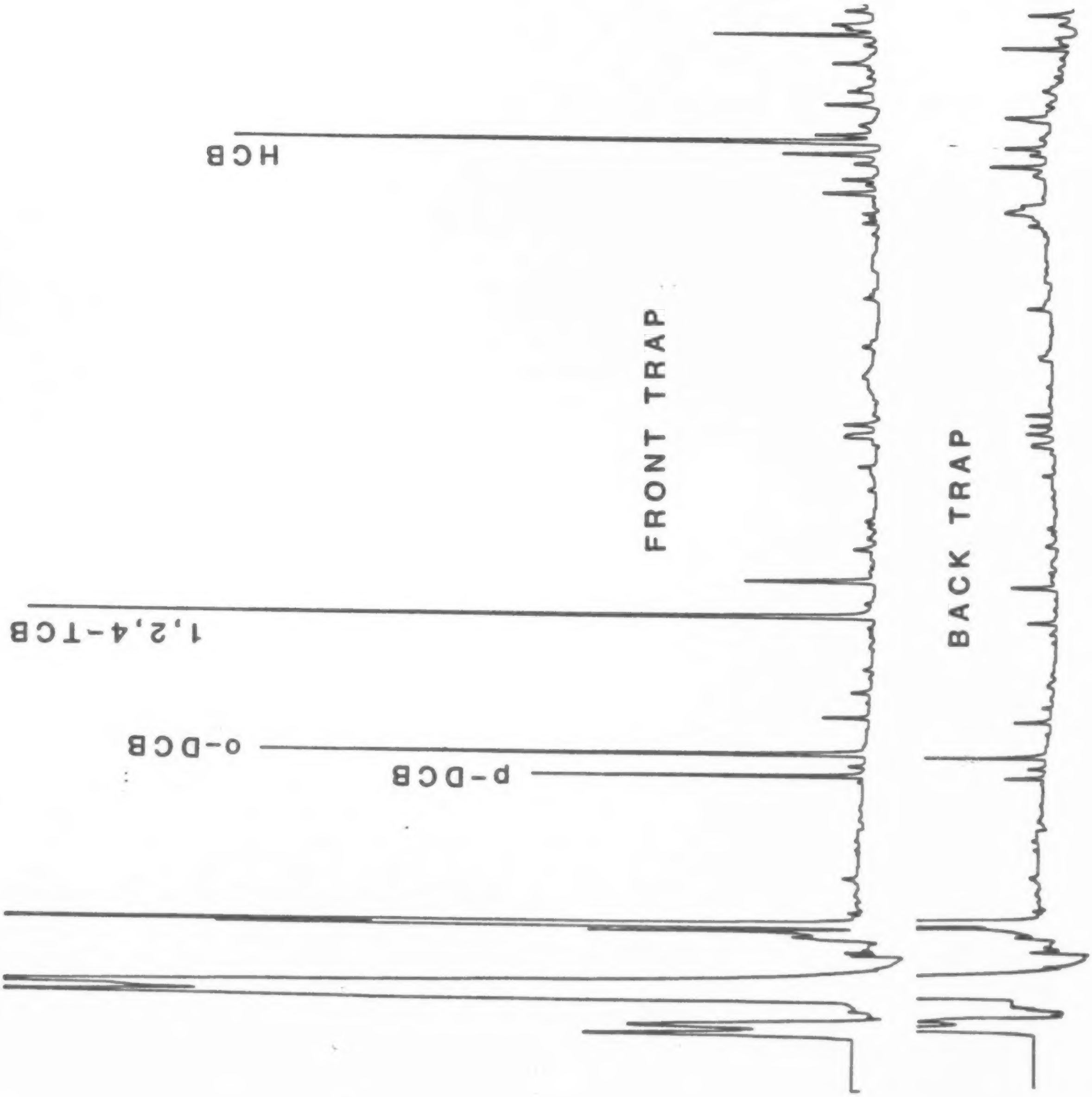


FIGURE 21. Front and back trap extracts from collection experiment 14 , 358 m^3 (Table A14). Standards are: A = m-DCB, B = p-DCB, C = o-DCB, D = 1,2,4-TCB, E = 1,2,4,5-TeCB, F = PeCB, G = α-HCH, H = HCB, I = γ-HCH. Note the poor collection of the DCBs at this air volume, and also the UCM at the late end of the front trap chromatogram.

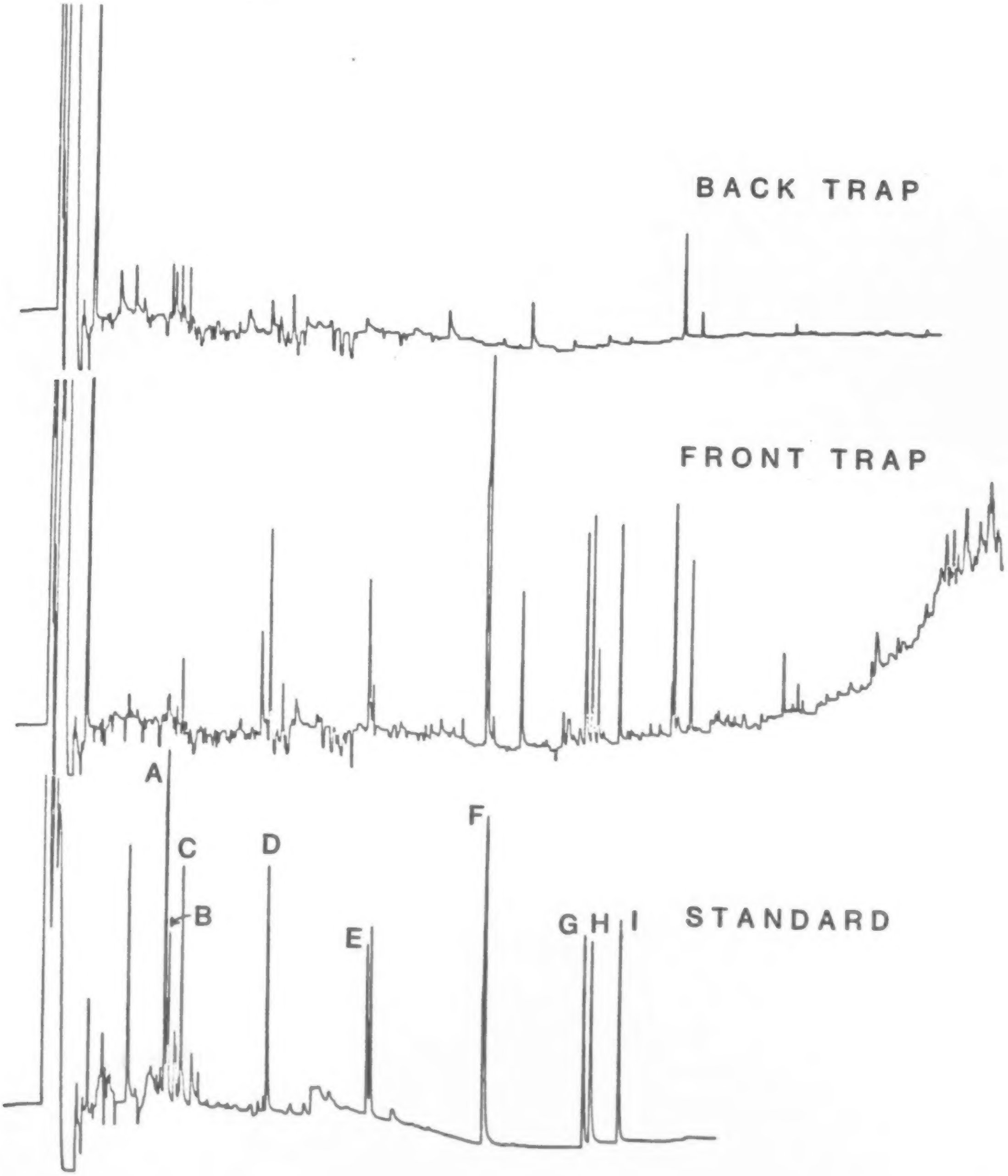
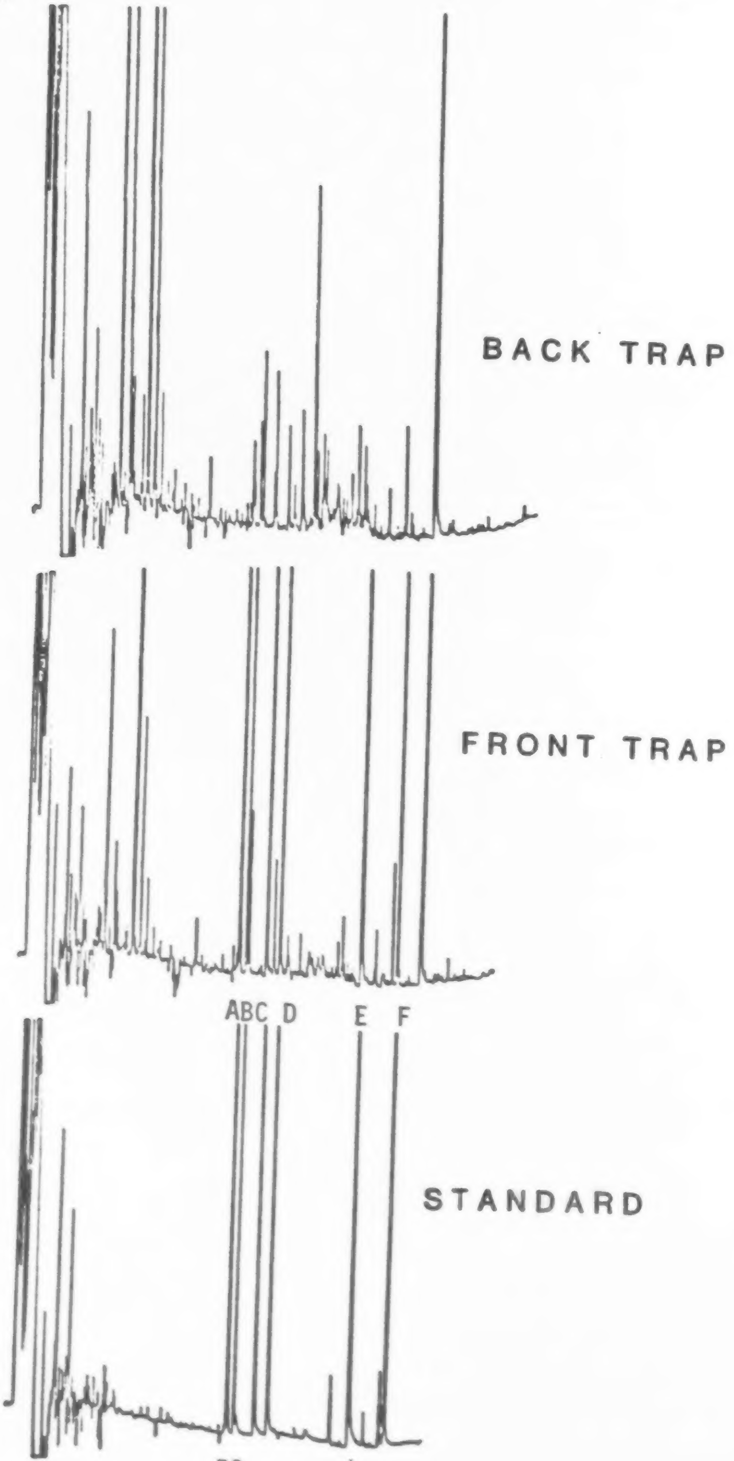


FIGURE 22. Front and back trap extracts from collection experiment 23, 334 m^3 (Table A23), chlorophenol fraction. A standard mixture of chlorophenol acetates is shown at the bottom; A = 2,6-DCP, B = 2,4-DCP, C = 3,4-DCP, D = 2,4,6-TCP, E = 2,3,4,5-TeCP, F= PCP. GC-ECD.



SECTION 4

RETENTION VOLUMES OF DICHLOROBENZENES ON PUF-TENAX CARTRIDGE.

An important step in the development of sampling methods for organic vapors is determining the collection efficiency of solid adsorbent cartridges. An adsorbent bed behaves as a frontal gas-solid chromatograph, in which solute is continuously introduced to and moved through the bed by the sampled air stream. The collection efficiency of an adsorbent cartridge for a particular solute is a function of the number of theoretical plates (N) in the bed and the breakthrough volume (V_B), defined here as the air volume when the solute concentration in the cartridge effluent is one-half the concentration introduced to the cartridge (Senum, 1981; You and Bidleman, 1984; Bidleman, 1985; Van Der Straeten et al., 1985).

It is experimentally easier to test adsorbent cartridges in an elution rather than a frontal mode. In elution chromatography, the solute is introduced to the cartridge as a pulse and then eluted as a band. The retention volume (V_R) is the volume of air required to elute the band maximum. Assuming linear adsorption isotherms, V_B for a frontal experiment and V_R for an elution experiment should be identical (Reilley et al., 1962).

Many V_R and V_B have been measured for volatile organic compounds such as solvent vapors, and N have been determined for small cartridges containing a few hundred milligrams of adsorbent such as those typically used in low volume sampling of workplace air (Butler and Burke, 1976; Gallant, 1978; Brown and Purnell, 1979; Pellizzari, 1977; Eiceman and Karasek, 1980; West, 1981; Clark and McIntyre, 1982; Van Der Straeten et al., 1985). These properties are usually determined by extrapolating gas chromatographic measurements at elevated temperatures to ambient temperatures. Less common are measurements of chromatographic properties for the larger adsorbent cartridges used in high volume (hi-vol) air samplers. These cartridges usually contain gram quantities of adsorbent, and sample volumes range from tens to thousands of cubic meters. Using hi-vol elution or frontal chromatography at ambient temperatures, we have determined V_R, V_B, and N for hexachlorobenzene (HCB), polychlorinated biphenyls (PCB) and polycyclic aromatic hydrocarbons (PAH) collected on cartridges packed with polyurethane foam (PUF)(Simon and Bidleman, 1979; Burdick and Bidleman, 1981; You and Bidleman, 1984; Bidleman et al., 1984; Bidleman, 1985;).

Compounds having liquid-phase saturation vapor pressures (p_L^o) above approximately 10^{-3} torr are not quantitatively collected by PUF under normal hi-vol sampling conditions. Lewis et al. (1982) showed that the low molecular weight polychlorinated biphenyls (PCB) were more completely collected by a sandwich cartridge, containing a granular adsorbent between two PUF slices, than with PUF alone. Adsorbents tested were Tenax-GC, Florisil, Chromasorb 102, and XAD-2. This study was undertaken to examine the chromatographic properties of a PUF-Tenax sandwich cartridge under hi-vol sampling conditions.

EXPERIMENTAL

The apparatus used for elution chromatography studies has been described in **Section 3** and shown in Figure 3. Briefly, the sampler was a PS-1 hi-vol (General Metal Works, Inc., Village of Cleves, Ohio) containing a PUF-Tenax sandwich cartridge. The PS-1 was modified by removing the standard 10-cm diameter filter holder and replacing it with a 12 x 15 x 100-cm stainless steel mixing chamber. Clean air entered the mixing chamber through a prefilter (20 x 25-cm glass fiber filter followed by an activated charcoal trap), which removed particles and organic contaminants from laboratory air. Test compounds in 25-50 μL methanol were injected through a septum into a heated glass port in the mixing chamber where they were vaporized, swept into the clean airstream, and onto the PUF-Tenax cartridge.

Separate front and back traps were used in the PS-1 (Figure 6). The front trap was an 8.0-cm x 5.8-cm i.d. glass cartridge packed with 10 g Tenax-GC (35/60-mesh) sandwiched between two 2.5-cm PUF slices, supported by a 10-mesh stainless steel screen. The back trap was a 5.0-cm x 5.8-mm i.d. glass cartridge containing 3 g Tenax held on a 1-cm PUF slice supported by a screen. A seal between the front and back cartridges was made with a red silicone rubber gasket. The overall dimensions of the two cartridges (12.5 cm x 5.8 cm i.d.) were the same as those of a standard PS-1 glass cartridge. Before use, the Tenax was soxhlet extracted overnight in paper thimbles with petroleum ether (PE) and dried under vacuum at 40°.

Experiments were carried out by vaporizing 200-300 μg dichlorobenzenes (DCBs) into the mixing chamber and pumping air through the apparatus at 0.20-0.22 m^3/min (linear velocity through cartridge 70-80 m/min). In this manner, the compound was introduced to the front trap as a vapor pulse which was then eluted through the cartridge by clean air. The back trap served as a detector for vapors eluting from the front trap, and was replaced after each 25-50 m^3 air. This frequent trap change (every 2 - 4 h) was necessary to define the elution curves. Test compounds were 1,2-, 1,3-, and 1,4-dichlorobenzenes (o-DCB, m-DCB, and p-DCB). Experiments were carried out in a room thermostated at 20°.

Tenax in the removed back traps was transferred to precleaned paper thimbles and soxhlet extracted with PE. At the end of each experiment, the PUF-Tenax front trap was extracted intact with PE in a specially designed soxhlet apparatus. Extract volumes were reduced by rotary evaporation and nitrogen blow-down, shaken with 7% fuming sulfuric acid to remove interfering electron-capturing compounds (chlorobenzenes are stable to this treatment), and analyzed by capillary gas chromatography (Varian 3700) with electron capture detection. Samples were injected in the splitless mode under the following conditions: column: 25-m BP-5 fused silica (SGE, Inc.), split time: 0.5 min, hydrogen carrier at 2 mL/min, oven program: 45° (5 min) increasing at 5°/min to 220°, injector: 220°, detector: 320°. Peak areas were integrated with a Hewlett-Packard 3390A or Shimadzu Chromatopac CR-3A instrument.

RESULTS AND DISCUSSION

The average DCB concentration in the effluent from the PUF-Tenax cartridge over a 2-4 h period was calculated by dividing the weight of DCB on a back trap by the air volume. Effluent concentrations were normalized to the maximum concentration during the run and were plotted against air volume to give elution chromatograms. An elution band of p-DCB is shown in Figure 23. The observed retention volume (V_R) from the peak maximum and the bandwidth at half height ($W_{1/2}$), as illustrated, were used to calculate N for the p-DCB band. We assume that V_R and N are properties of the Tenax layer, since separate analyses of the top PUF slice in the front trap revealed negligible retention of the DCBs.

Although similar experiments with PCB, HCB, and PAH on PUF alone gave reasonably symmetric elution bands or fronts (Simon and Bidleman, 1979; Burdick and Bidleman, 1981; You and Bidleman, 1984; Bidleman et al., 1984; Bidleman, 1985), only p-DCB gave a well-shaped band on PUF-Tenax. Some tailing was observed for m-DCB, and the tailing was most severe for o-DCB (Figure 24). In the case of o-DCB, the band maxima were poorly defined in two experiments, and thus the observed V_R varied from 80-140 m^3 (Table 29). The N values calculated for p-DCB as shown in Figure 23 ranged from 3.3-3.9 (mean = 3.6) in three experiments. Because the m-DCB and o-DCB bands were asymmetric, N values for these compounds were not calculated.

Gas chromatographic retention times on the BP-5 column follow the order m-DCB < p-DCB < o-DCB, decreasing with increasing P_L^o (Table 1). Since V_R or V_B of organochlorines and PAH on PUF were inversely correlated to P_L^o (Bidleman et al., 1984; You and Bidleman, 1984; Bidleman, 1985), we expected the V_R of the DCBs on PUF-Tenax to be: m-DCB < p-DCB < o-DCB. The observed V_R order from band maxima was: o-DCB < m-DCB < p-DCB (Figure 24).

The anomolous behavior of o-DCB may be an artifact of the band asymmetry. Conder et al. (1983, 1986) discussed the problem of determining V_R for skewed bands in gas-liquid and gas-solid chromatography. The V_R at infinite dilution is underestimated for tailing bands. Several methods of calculating V_R from skewed bands were examined by Conder et al., and one of the more satisfactory ones was:

$$V_R = (V_I + V_F)/2 \qquad \text{Equation 1}$$

where V_I and V_F are the retention volumes or times defined by the points where tangents to the leading and trailing edges of the band at the points of inflection intersect the baseline. This relationship provides a good estimate of the band center of mass (Conder et al., 1983). If Equation 1 is applied to the bands in Figure 24 (taking $V_I = 0$), the calculated V_R are: m-DCB = 230 m^3, p-DCB = 250 m^3, and o-DCB = 260 m^3. Only for the most symmetric band (p-DCB) is this calculated V_R close to the V_R observed from the band maximum. The calculated V_R for each experiment are also given in Table 29, and are in the order expected from the p_L^o of the three DCBs.

Retention volumes per gram Tenax (V_g), based on observed or calculated V_R and a 10-g load of Tenax in the front trap, are given in Table 29. Many V_g have been published for volatile compounds such as solvent vapors, but only a few V_g have been determined for halobenzenes: mono- through trichlorobenzene, bromobenzene, and benzyl chloride (Pellizzari, 1977; Gallant, 1978; Brown and Purnell, 1979; Eiceman and Karasek, 1980; West, 1981). V_g for p-DCB in this work agrees excellently with one reported value (Gallant, 1978), and within a factor of two with the V_g from another study (Eiceman and Karasek, 1980). However the V_g of m-DCB and o-DCB are much higher than those determined by Pellizzari (1977). A log-log plot of V_g vs. p_L^o for halobenzenes shows the expected inverse relationship (Figure 25), but the disagreement among reported V_g for the same compound is very large, often about a factor of ten. Various determinations of V_g fall into high and low groups, with ours in the high category. It is interesting that in this work and the study of Pellizzari (1977) the ratio of observed V_R for m-DCB/o-DCB is 1.5, despite a factor of 20 difference in the absolute magnitude of the observed V_R values in the two studies.

To simulate an air sampling situation, o-DCB and p-DCB were injected into the mixing chamber and collected on a train consisting of front and back PUF-Tenax cartridges (Figure 6) or a "double sandwich" cartridge containing 10-g front and 3-g back Tenax layers separated by PUF slices (Figure 5). Quantities of DCBs used in these experiments were 2-40 µg, about 1-10% of those used for the elution chromatography experiments, and were in the range of the µg DCBs that would be collected by sampling 30-100 m^3 ambient air (Harkov et al., 1985; Wallace et al., 1985). Front and back cartridges (separate traps) or Tenax layers (double sandwich cartridges) were analyzed individually after drawing an air sample volume (V_S) of 30-165 m^3 through the system.

The distribution of DCBs within the sampling train and overall collection efficiencies determined by mass balance are given in Table 30. Total recovery of the DCBs was good, averaging 89% of quantities injected into the mixing chamber for V_S up to 165 m^3. Quantitative collection of p-DCB was expected, since its observed V_R (220 m^3) is well above V_S. However o-DCB also showed little breakthrough, even for experiments in which V_S equalled or exceeded the observed V_R from elution experiments (97 m^3).

As discussed earlier, the observed V_R of o-DCB from the elution experiments may be anomolously low because of the band asymmetry. The calculated V_R of the three DCBs are all above 200 m^3. The o-DCB elution profile at the lower vapor concentrations employed in the air sampling simulation experiments has not been determined. If it is more symmetric, the calculated V_R would more accurately represent the collecting ability of the cartridge. This implies that Tenax was overloaded with o-DCB in the elution chromatography experiments. However quantities of o-DCB and p-DCB in the elution experiments were about the same, and the p-DCB band was reasonably symmetric (Figures 23 and 24). Further work would be required to determine whether the concentration ranges over which adsorption isotherms are linear differ for o-DCB and p-DCB. Even if the o-DCB band shape at low vapor concentrations are the same as that shown in Figure 24, the tailing would result in greater retention on the front trap than would be expected from its observed V_R.

All laboratory experiments in this study were carried out in the elution mode. Collection efficiencies under the frontal chromatography situation of actual air sampling can be predicted from V_R and N, assuming a gaussian profile of the solute front (Senum, 1981; You and Bidleman, 1984; Bidleman, 1985; Van Der Straeten et al., 1985). Of the three DCBs collected on Tenax, only p-DCB approximately fulfills this condition (Figures 23 and 24). Senum (1981) and Van Der Straeten (1985) have presented convenient nomographs relating V_S/V_R, N, and the fraction collected. At V_S = 100-150 m^3, V_S/V_R = 0.45-0.68 for p-DCB. Using Senum's nomograph at N = 3.6, 85-90% of the p-DCB should be trapped by the front PUF-Tenax cartridge. To check the collection efficiency of the train under field conditions, two ambient air samples of 110 and 150 m^3 were taken in the city of Columbia, SC using front and back PUF-Tenax sandwich cartridges. Concentrations of p-DCB and o-DCB were 26-84 ng/m^3 and 8-25 ng/m^3, respectively. The fraction of p-DCB found in the front trap was 94-96%. Collection of o-DCB was also quantitative, (91-92% on front trap), despite the fact that V_S > the observed V_R = 97 m^3. However if the calculated V_R = 250 m^3 is the more correct chromatographic parameter, the predicted collection efficiency of o-DCB is similar to that of p-DCB and agrees with field results.

TABLE 29. Retention Volumes and Specific Retention Volumes for Dichlorobenzenes on a PUF-Tenax Cartridge[1].

	P_L^o	V_R, m^3		V_g, m^3/g		Literature V_g
	torr, 20°	Obs[2]	Calc[3]	Obs	Calc	m^3/g
m-DCB	1.69	125	230	12.5	23	0.62 (4)
p-DCB	1.45	200	205			
		220	250			
		240	250			
	avg.	220	235	22	23.5	17 (5), 9.4 (6)
o-DCB	1.06	80-100	210			
		80-140	280			
		90	260			
	avg.	97	250	9.7	25	0.42 (4)

---

1. Three experiments were run with o-DCB and p-DCB, and one with m-DCB. Quantities of DCBs used were 200-300 μg.
2. Observed from band maxima.
3. Calculated from Equation 1.
4. Pellizzari, 1977.
5. Gallant, 1978.
6. Eiceman and Karasek, 1980.

TABLE 30. Collection Efficiencies of DCBs on a PUF-Tenax Cartridge in Air Sampling Simulation Experiments[1].

	Experiments	% on Front Trap[2]	% Recovery[3]
p-DCB			
32-34 m^3	2	96-97	99-101
78 m^3	1	99	63
100-125 m^3	3	92-94	76-90
165 m^3	1	89	88
o-DCB			
32-24 m^3	2	96-99	88-102
78 m^3	1	94	106
100-125 m^3	3	86-94	83-93
165 m^3	1	89	100

---

1. 2-40 µg DCBs used in these experiments.
2. 100[µg front/(µg front + µg back)]
3. % of quantities injected into mixing chamber, front + back traps, corrected for 86% analytical recovery.

75

FIGURE 23. Elution band of p-DCB from PUF-Tenax sandwich cartridge.

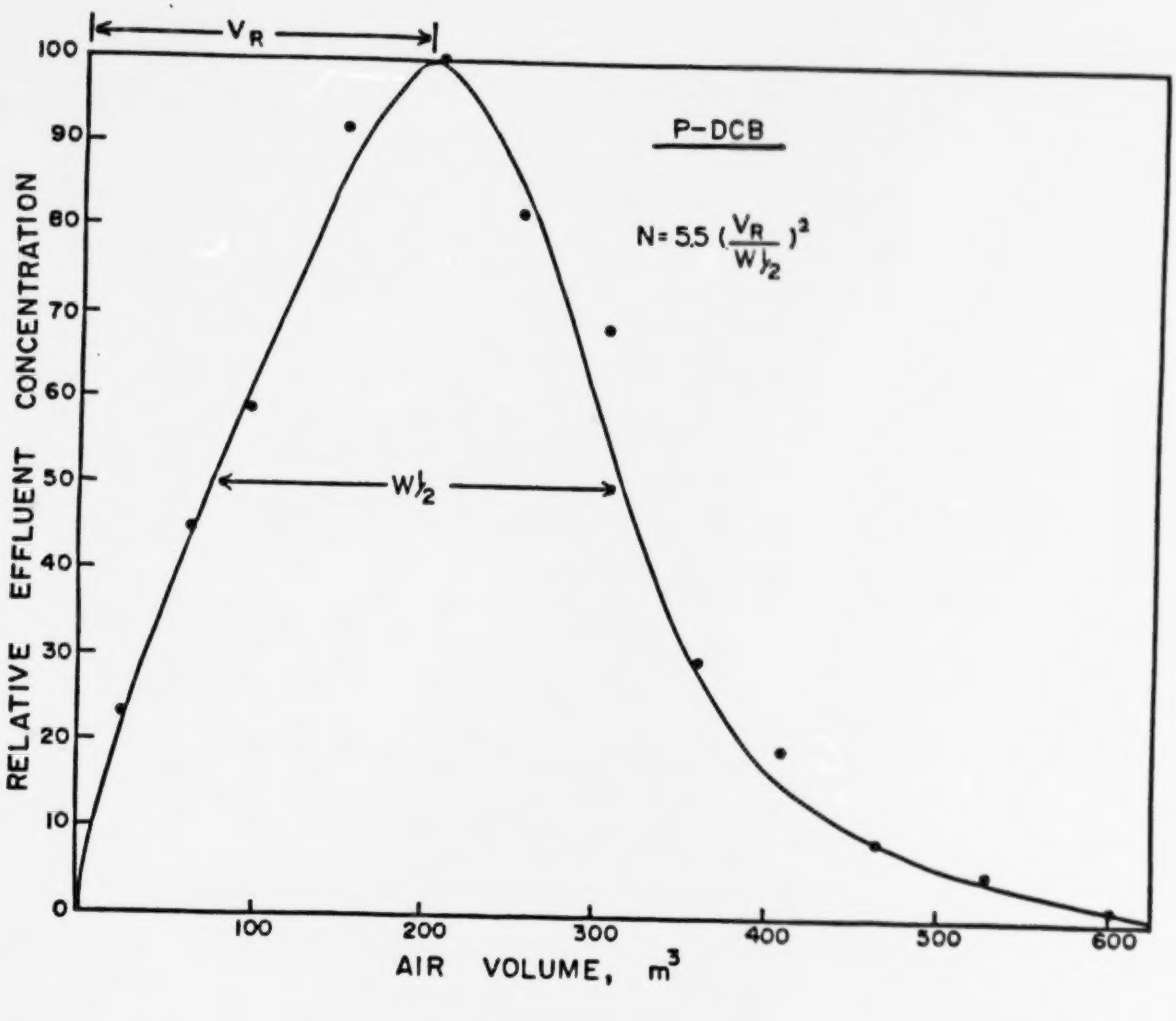


FIGURE 24. Elution bands of three DCBs injected in a single experiment.

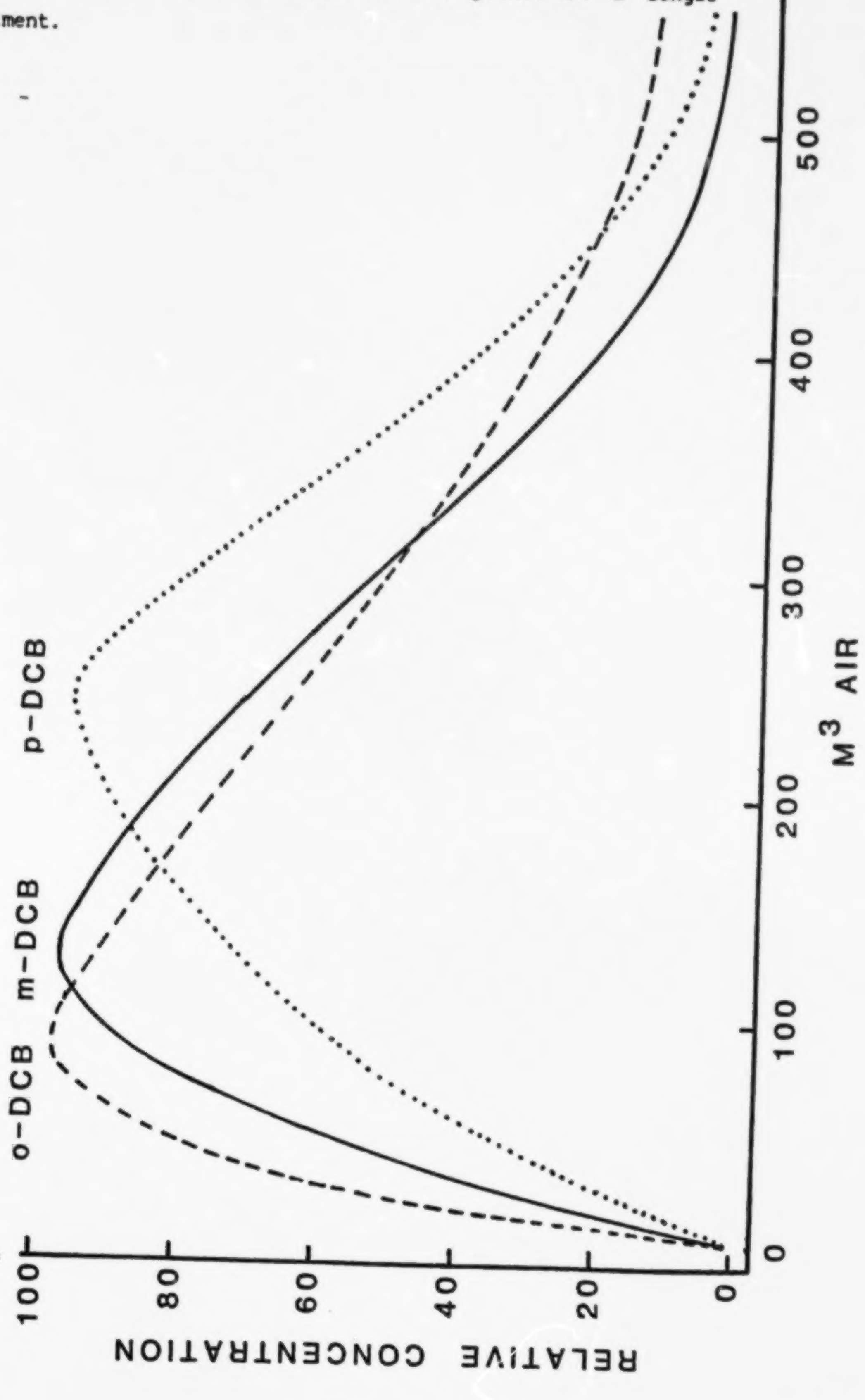


FIGURE 25. Relationship of specific retention volume (V_g) of halobenzenes on Tenax to liquid-phase vapor pressure at 20° (p_L^o, torr). Bars define the range of V_g in this work from observed or calculated V_R. Literature V_g (Pellizzari, 1977; Gallant, 1978; Eiceman and Karasek, 1980; West, 1981) are shown by circles.

1 = chlorobenzene

2 = bromobenzene

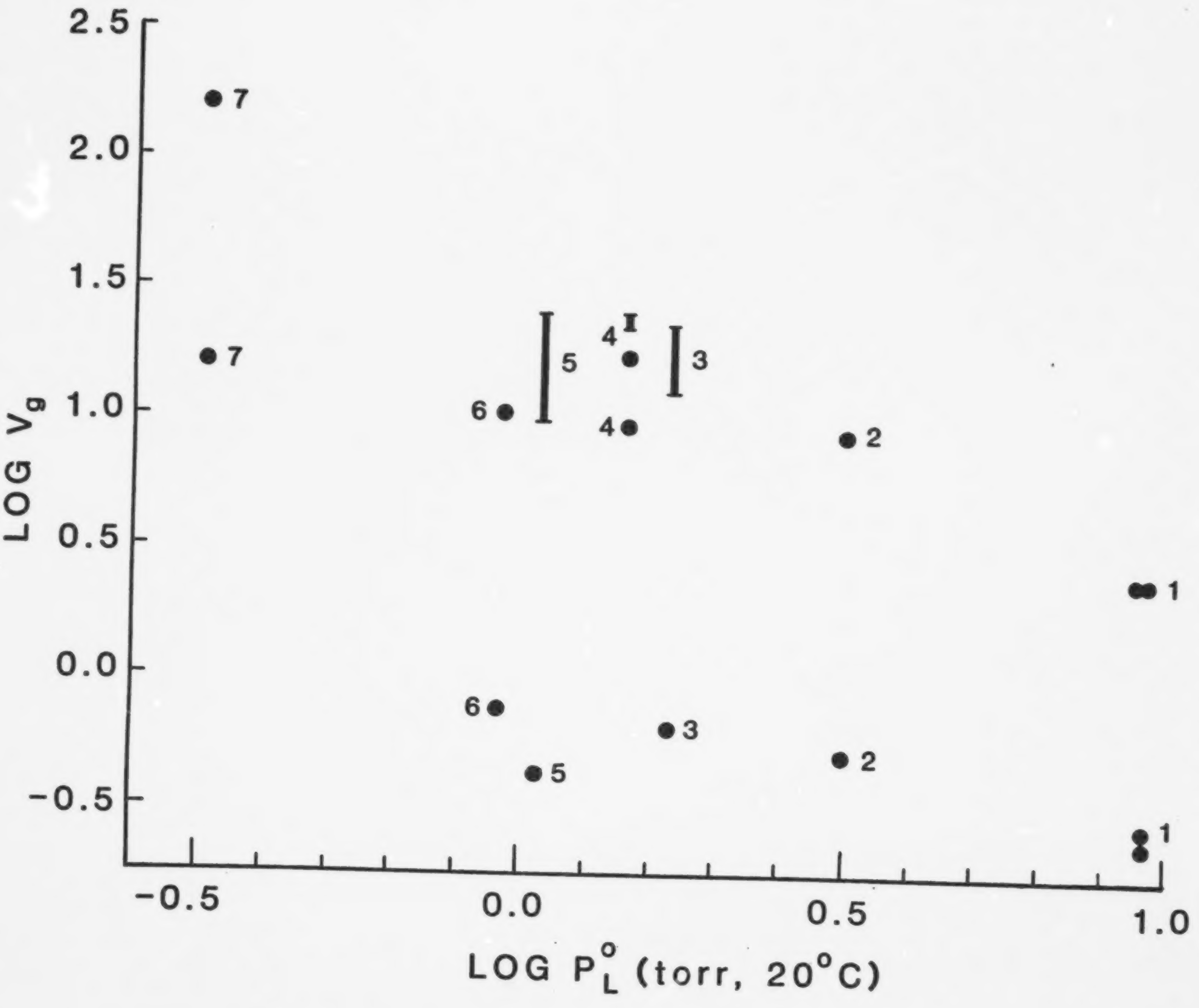
3 = m-dichlorobenzene

4 = p-dichlorobenzene

5 = o-dichlorobenzene

6 = benzyl chloride

7 = 1,2,4-trichlorobenzene


2.5
2.0
1.5
1.0
0.5
0.0
−0.5
LOG V_g
−0.5
0.0
0.5
1.0
LOG P_L^o (torr, 20°C)
7
7
6
5
4
4
3
2
1
6
3
5
2
1

SECTION 5

AMBIENT AIR SAMPLING

To assess the collection capabilities of a PUF-Tenax sandwich cartridge under field conditions, ambient air samples were taken at an urban and rural site. Collections in the city of Columbia, SC (pop. 300,000) were made September 19-20, 1985 (night) and December 12, 1985 (day) on the roof of the Byrne International Center, a 7-story building on the University of South Carolina campus. Rural samples were taken at North Inlet estuary, near Georgetown, SC, on November 22-23, 1986 (day and night).

EXPERIMENTAL

Collections were made using the PS-1 containing front and back PUF-Tenax traps. The tandem cartridges in Figure 6, with the front trap containing 10 g Tenax, were used for sampling in 1985. The separate front and back sandwich cartridge system in Figure 7, with 5 g Tenax in each trap, was used in 1986. Collection details are given in Tables 31-34.

Analytical procedures were as described in Section 3 Cartridges were extracted with PE (Sept., 1985) or 15% EE-PE (Dec. 1985, Nov. 1986). Filters were cut into strips and refluxed with dichloromethane (DCM). The DCM was replaced with hexane during subsequent concentration steps. Extracts were concentrated by flash evaporation and nitrogen blow-down; the Dec. 1985 and Nov. 1986 samples were partitioned with aqueous base to separate chlorophenols, which were then derivatized to acetates and chromatographed as previously described.

Base-neutral extracts were fractionated to separate chlorobenzenes and PCB from organochlorine pesticides. Our procedure was adapted from the one given by Keller and Bidleman (1984). Silicic acid (Mallinckrodt, 200-mesh) was dried at 140°, 3-g portions were weighed into vials, and the vials were returned to the oven until needed. A vial was removed from the oven, capped, and allowed to come to room temperature. Distilled water (0.15 mL) was added and the vial contents were mixed thoroughly. After equilibrating for an hour or longer, the silicic acid (containing 5% added water) was dry-packed into a 1-cm i.d. Chromaflex column. The silicic acid layer was overlaid with 2 g neutral alumina (containing 6% added water), and topped with 1 cm anhydrous sodium sulfate. The column was pre-washed with 30 mL DCM or 30% DCM-PE, followed by 30 mL PE. The sample was added in 2-3 mL hexane or PE, and the column was eluted with 25 mL PE (Fraction 1) followed by 25 mL 30% DCM-PE (Fraction 2). Fractions were concentrated into isooctane by nitrogen blow-down and treated with concentrated sulfuric acid or 7% fuming sulfuric acid before GC-ECD analysis. Acid treatment was omitted in the analysis of PAH or oxygen-containing pesticides (e.g., dieldrin, endosulfan).

In the above scheme, Fraction 1 contains: n-alkanes, most of the UCM from atmospheric organic matter (Keller and Bidleman, 1984), chlorobenzenes, PCB, p,p'-DDE, and certain components of technical chlordane (chlordene isomers). Fraction 2 contains: PAH, p,p'-DDT, p,p'-DDD, cis- and trans-chlordane, trans-nonachlor, HCHs, dieldrin, endosulfan, and toxaphene. Heptachlor and o,p'-DDT divide between the two fractions.

Samples were quantitatively analyzed by GC-FID (Sept. 1985) and GC-ECD (Dec. 1985, Nov. 1986), using the columns and conditions described previously, and qualitatively by gas chromatography - mass spectrometry (GC-MS) in the electron impact (EI) and negative ionization (NI) modes. These analyses were done by Dr. Michael D. Walla on the Chemistry Department's Finnigan 4521C instrument, using a 25-m DB-5 (J & W Corp.) or equivalent column. Samples were injected in the splitless mode under the following conditions: 2 mL/min He carrier gas, inject at 50^{o}, hold 0.1 min, then program at 5^{o}/min to 300^{o}. In the negative ionization mode, 0.25 torr methane was used. Runs were made in the multiple ion detection (MID) mode, monitoring the ions given in Table 35.

RESULTS AND DISCUSSION

Collection data and analytical results for the four air samples are presented in Tables 31-34. Only the September, 1985 sample was analyzed by GC-FID for n-alkanes and 2-ring aromatic hydrocarbons. A clear pattern of n-C_{11} through n-C_{23} alkanes was found on the front trap, with only traces found on the back trap (Figure 26). Absent from this chromatogram is the UCM generally found in fraction 1 of urban air extracts (Keller and Bidleman, 1984; Farmer and Wade, 1986). Four 2-ring aromatic hydrocarbons were identified in fraction 2 by retention times: NAP, 2-MN, 2,6-DMN, and BiPH. However the chromatogram showed some very large, tailing peaks (Figure 27), and it was obvious that these aromatic hydrocarbons were not the main sample components. Fraction 2 SOC were collected effectively; chromatograms of front and back traps showed very little breakthrough (Figure 27).

Several chlorobenzenes were identified by GC-ECD in September and December air samples from Columbia (Tables 31 and 32); an example chromatogram is shown in Figure 28. Breakthrough of dichlorobenzenes to back traps was $\leq$10% of front-trap quantities. This is in agreement with laboratory collection data (Tables 20, 21, 27), which indicated quantitative trapping in the air volume range of these samples (108-148 m^3).

Front and back traps from the December 1985 sample were examined for chlorobenzenes by EI and NI GC-MS, using the MID technique and the ions in Table 35. Two dichlorobenzenes (o-DCB and p-DCB) were found by EI, and the chromatograms again showed their good collection efficiency (Figure 29). The additional sensitivity of NI was needed to see PeCB and HCB, which were clearly identified on the front trap. Penetration to the back trap was negligible (Figure 30).

ECD chromatograms of heavier organochlorines in the December, 1985 air sample are shown in Figures 31 and 32. The fraction 1 chromatogram showed a PCB pattern on the front trap and a clean back trap and blank (Figure 31). Two HCHs (α-HCH and γ-HCH) and chlordanes (cis- and trans-chlordane, trans-nonachlor) were found in fraction 2 (Figure 32). However, the later portion of the front trap chromatogram was obscured by a UCM, the origin of which is uncertain. Its presence has been discussed in section II-2C.

Fraction 1 and 2 organochlorine levels at North Inlet estuary are given in Tables 33 and 34, and example chromatograms are shown in Figures 33 and 34. A substantial artifact resembling o-DCB was found on filters from these runs. Given the volatility of o-DCB, we do not think that it is actually filter-retained. A blank PUF-Tenax cartridge was taken into the field during this study, stored 4 months (frozen), and analyzed. It was clean, except for three large peaks, one of which overlapped 1,2,4-TCB (Figure 13).

Tetrachlorobenzene and heavier compounds showed good collection efficiencies at North Inlet, as indicated by low breakthrough. Breakthrough was also not too high for 1,2,4-TCB (Tables 33 and 34). These field results confirm laboratory findings that a PUF-Tenax cartridge containing 5 g Tenax can effectively collect tetrachlorobenzene and less volatile SOC from up to 360 m^3 air.

Comparisons of SOC levels in this study with those in other locations are made in Tables 36-38. The C_{20} - C_{22} n-alkanes in the one Columbia sample were higher than those found in two other cities. On the other hand, NAP and 2-MN were near the low end of the range in Portland, OR (Table 36). Considering that only a single sample in Columbia was analyzed for hydrocarbons, the values in Table 36 do not appear to be unreasonable.

The range of dichlorobenzenes among cities is very large, and ours fall near the low end (Table 37). HCB is one of the more uniform organochlorines in the troposphere, as can be seen from the narrow range of concentrations in Table 37. Our HCB and PeCB concentrations compare very well with other reported levels.

Concentrations of the HCHs in this study fell within the range of those in other localities, although γ-HCH at North Inlet seems low (Table 38). PCB and chlordane levels were determined only in Columbia, and fell within the range of concentrations previously measured from 1977-80 (Billings and Bidleman, 1980, 1983; Table 38).

Ambient air samples 2-4 were analyzed for chlorophenols by GC-ECD after derivatization to the acetates. The resulting chromatograms were very complex, and the major peaks did not match the retention times of our chlorophenol standards (Figures 35 and 36). The quantitative results in Tables 32-34 should be considered upper limits only. It seems likely that many other acidic compounds in the atmosphere could be derivatized and detected by GC-ECD. Examples are nitrophenols, chloroguaiacols, and chlorocatechols. Further work with ambient chlorophenols will require a more selective analytical technique than GC-ECD, and we will be using MID GC-MS in future studies.

TABLE 31. Ambient Air Sample 1, Columbia, SC, Sept. 19-20, 1985.

Air Volume: 148 m^3

Temperature: 20-30°C

Normal Alkanes

Carbon #	ug Front Trap	ug Back-up Trap[a]	ug/m^3
12	17.8	0.15	0.12
13	8.9	ND	0.060
14	4.5	0.15	0.030
15	5.2	0.37	0.033
16	4.4	0.59	0.026
17	3.3	0.46	0.020
18	2.2	0.16	0.014
19	1.9	0.22	0.012
20	1.8	0.47	0.0091
21	2.1	0.53	0.010
22	2.1	0.20	0.013
23	1.4	0.36	0.0072
24	0.72	0.65	—
sum			0.34

Organochlorines	ng Front Trap	ng Back-up Trap	ng/m^3
p-DCB	1.2×10^4	4.4×10^2	87
o-DCB	1.3×10^3	1.1×10^2	9.6
1,2,4-TCB	880	10	6.0
PeCB	6	ND	0.04
HCB	27	ND	0.18
heptachlor	122	ND	0.82
p,p'-DDE	11	ND	0.075
Aroclor 1016	299	ND	2.0
Aroclor 1254	428	ND	2.8
alpha-HCH	153	1	1.0
gamma-HCH	52	0.5	0.35
trans-chlordane	113	1.6	0.75
cis-chlordane	60	1.2	0.38
trans-nonachlor	39	0.9	0.26

2-ring PAH	ug Front Trap	ug Back-up Trap	ug/m^3
biphenyl	1.7	0.28	0.0095
naphthalene	27	ND	0.18
2-methylnaphthalene	16	ND	0.11
2,6-dimthylnaphthalene	4.3	ND	0.028

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[a] Back-up Trap quantities used as blanks for n-alkanes in Front Trap

TABLE 32. Ambient Air Sample 2, Columbia, SC, December 12, 1985.

Air Volume: 108 m^3

Temperature: 19-20°C

Organochlorines	ng Front Trap	ng Back-up Trap	ng blank	ng/m^3
p-DCB	2.6×10^3	1.8×10^2	1.9×10^1	23
o-DCB	2.5×10^3	2.5×10^2	1.2×10^2	24
1,2,4-TCB	23	2	1.7	0.21
PeCB	13	0.6	0.5	0.13
HCB	14	0.4	0.4	0.13
heptachlor	87	7	ND	0.87
Aroclor 1016	570	53	53	4.8
Aroclor 1254	392	32	26	3.4
alpha-HCH	28	0.8	0.6	0.26
gamma-HCH	13	1.7	1.7	0.10
trans-chlordane	26	4.4	2.8	0.21
cis-chlordane	13	2.3	1.4	0.11
trans-nonachlor	9.5	1.8	1.0	0.078
Chlorophenols[1]				
2,6-DCP	242	33	ND	2.5
2,4-DCP	303	ND	ND	2.8
3,4-DCP	40	6.4	ND	0.42
2,4,6-TCP	39	12.5	1.0	0.47
2,3,4,5-TeCP	ND	ND	ND	ND
PCP	9.8	5.1	3.5	0.14

---

1. Upper limits only.

TABLE 33. Ambient Air Sample 3, North Inlet Estuary, SC, November 22, 1986.

Air volume: 360 m^3

Compound		ng Found[a]		
	filter	front	back	ng/m^3
Organochlorines:				
m-DCB	<12.8	<422.6	<165.4	-
p-DCB	<23.5	4260.0	622.0	13.6
o-DCB	111.1	98.3	<310.6	0.27
1,2,4-TCB	9.6	2874.0	<210.0	8.0
1,2,4,5-TeCB	<0.73	31.8	3.5	0.098
PentaCB	<0.22	18.2	<0.93	0.051
Hexa-CB	1.6	32.8	<1.3	0.096
alpha-HCH	<0.33	17.5	<2.0	0.049
gamma-HCH	1.1	6.8	<0.94	0.019
Chlorophenols:[b]	filter	front	back	ng/m^3
2,6-DCP	15.8	196.4	55.7	0.74
2,4-DCP	2.79	25.5	4.63	0.091
3,4-DCP	6.08	45.9	52.3	0.29
2,4,6-TCB	0.96	4.37	<.93	0.015
2,3,4,5-TeCP	3.82	1.13	<.38	0.014
Penta-CP	23.9	16.0	7.01	0.13

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a) Organochlorines were corrected for the average operational blank of the laboratory collection experiments, but the chlorophenols are uncorrected.

b) Upper limits only.

TABLE 34. Ambient Air Sample 4, North Inlet Estuary, SC, November 23, 1986.

Air volume: $330\ m^3$

Compound	ng Found[a]			
	filter	front	back	ng/m^3
Organochlorines:				
m-DCB	<11.3	<190.5	<136.3	–
p-DCB	<19.7	1358.0	1052.7	7.3
o-DCB	338.5	<318.2	<415.2	–
1,2,4-TCB	<1.1	436.3	<137.4	1.3
1,2,4,5-TeCB	3.03	<11.1	<18.1	–
PentaCB	<0.2	8.0	<0.77	0.024
Hexa-CB	0.28	35.2	<1.52	0.11
alpha-HCH	1.13	42.6	<1.25	0.13
gamma-HCH	<0.25	3.77	<0.29	0.011
Chlorophenols:[b]	filter	front	back	ng/m^3
2,6-DCP	<1.5	6.26	2.58	0.27
2,4-DCP	<1.6	31.1	3.95	0.11
3,4-DCP	5.38	18.7*	<1.1	0.073
2,4,6-TCB	<.4	10.8**	1.45	0.037
2,3,4,5-TeCP	0.5	0.46	0.80	0.005
Penta-CP	2.8	5.32	13.8	0.066

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* Peak was resolved in only 1 of 3 injections.

** Peak was outside of retention time window.

a) OC's corrected for average operational blank of lab exp. CP's are uncorrected for operational blanks.

b) Upper limits only.

TABLE 35. Ions Monitored for GC-MS Confirmation of Chlorobenzenes.

Electron Impact Mode	
dichlorobenzenes	146, 148, 150
trichlorobenzenes	180, 182, 184
tetrachlorobenzenes	214, 216, 218
Negative Ionization Mode	
pentachlorobenzene	248, 250, 252
hexachlorobenzene	282, 284, 286

TABLE 36. Comparison of Vapor-Phase Hydrocarbons in Columbia, SC and Other Locations ($\mu g/m^3$).

Alkanes	Columbia, SC Sept. 1985	Portland, OR[1] 1984	Dourbes, Belgium[2] 1979
C_{20}	9.1	2.2-6.7	3.7-6.3
C_{21}	10	0.8-3.2	1.9-4.4
C_{22}	13	0.9	
Aromatic Hydrocarbons			
NAP	180	280-940	
2-MN	110	110-460	

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1. Ligocki et al., 1985.

2. Van Vaeck et al., 1984.

TABLE 37. Comparison of Vapor-Phase Chlorobenzenes in Columbia, SC, North Inlet, SC, and Other Locations (ng/m^3).

	o-DCB	m-DCB	p-DCB	1,2,4-TCB	PeCB	HCB
Columbia, SC Sept.-Dec. 1985	10-23		2-87	0.2-6.0	0.04-0.13	0.13-0.18
Columbia, SC 1977-80 (1)						0.19-0.40
North Inlet, SC Nov. 1986	1.0-2.5	0.6-1.2	7-15	2.0-8.6	0.04-0.06	0.10-0.11
Portland, OR 1984 (2)	3.3-10		73-170			0.05-0.11
Denver, 1980 (1)						0.19-0.30
U.S. median (3)	66		280[4]			
NJ median (3)	170		1000[4]			
College Station, TX (5)						0.14-0.39
Lillestrøm, Norway, 1980 (6)					0.03-0.08	0.06-0.23

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1. Billings and Bidleman, 1980, 1983.

2. Ligocki et al., 1985.

3. Wallace et al., 1985.

4. m-DCB + p-DCB.

5. Atlas and Giam, 1987.

6. Oehme and Ottar, 1984.

TABLE 38. Comparison of Vapor-Phase Organochlorines in Columbia, SC, North Inlet, SC, and Other Locations (ng/m^3).

	α-HCH	γ-HCH	Aroclor 1242	Aroclor 1254	Chlordane[1]
Columbia, SC Sept.-Dec. 1985	0.26-1.0	0.26-0.35	2.0-4.8	2.8-3.4	0.40-1.4
Columbia, SC 1977-80 (2)			1.6-5.7	0.56-3.8	0.38-3.2
North Inlet, SC Nov. 1986	0.07-0.15	0.02-0.03			
Denver, 1980 (2)	0.21-0.43[3]		1.1-2.2	0.08-0.36	0.013-0.069
College Station, TX, (4)	0.09-2.3	0.01-1.6		0.11-0.48	0.32-2.6
Minneapolis, MN, 1978-79 (5)			1.0-15	0.17-2.6	
Chicago, IL 1975-76 (6)			2.9-10	0.34-2.4	
Madison, WI, 1978 (7)			5.7-6.8	1.0	
Lake Michigan, 1977 (7)			0.35-0.88	0.09-0.45	
Lake Superior, 1978-80 (5)			0.07-2.5	0.03-1.0	
Portland, OR 1984 (8)	0.29-0.44				
Lillestrøm, Norway (9)	0.35-1.2	0.15-0.41			

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1. cis- + trans-chlordane + trans-nonachlor
2. Billings and Bidleman, 1980, 1983.
3. α- + γ-HCH.
4. Atlas and Giam, 1987
5. Eisenreich et al., 1983.
6. Murphy et al., 1978.
7. Doskey and Andren, 1981.
8. Ligocki et al., 1985.
9. Oehme and Ottar, 1984.

FIGURE 26. Ambient air sample 1 from Columbia, SC (September, 1985), fraction 1. GC-FID.

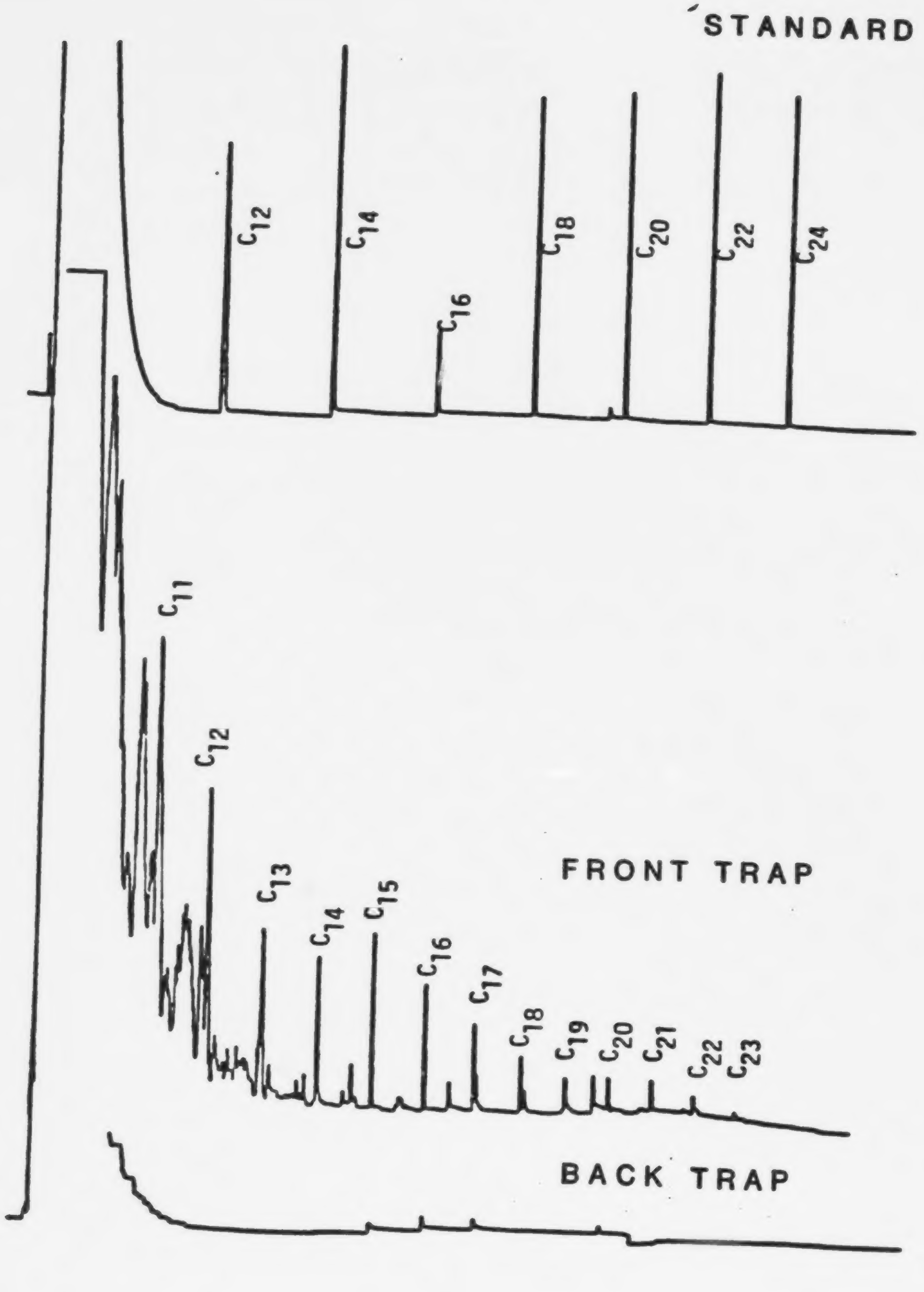


FIGURE 27. As in Fig. 26, fraction 2. GC-FID.

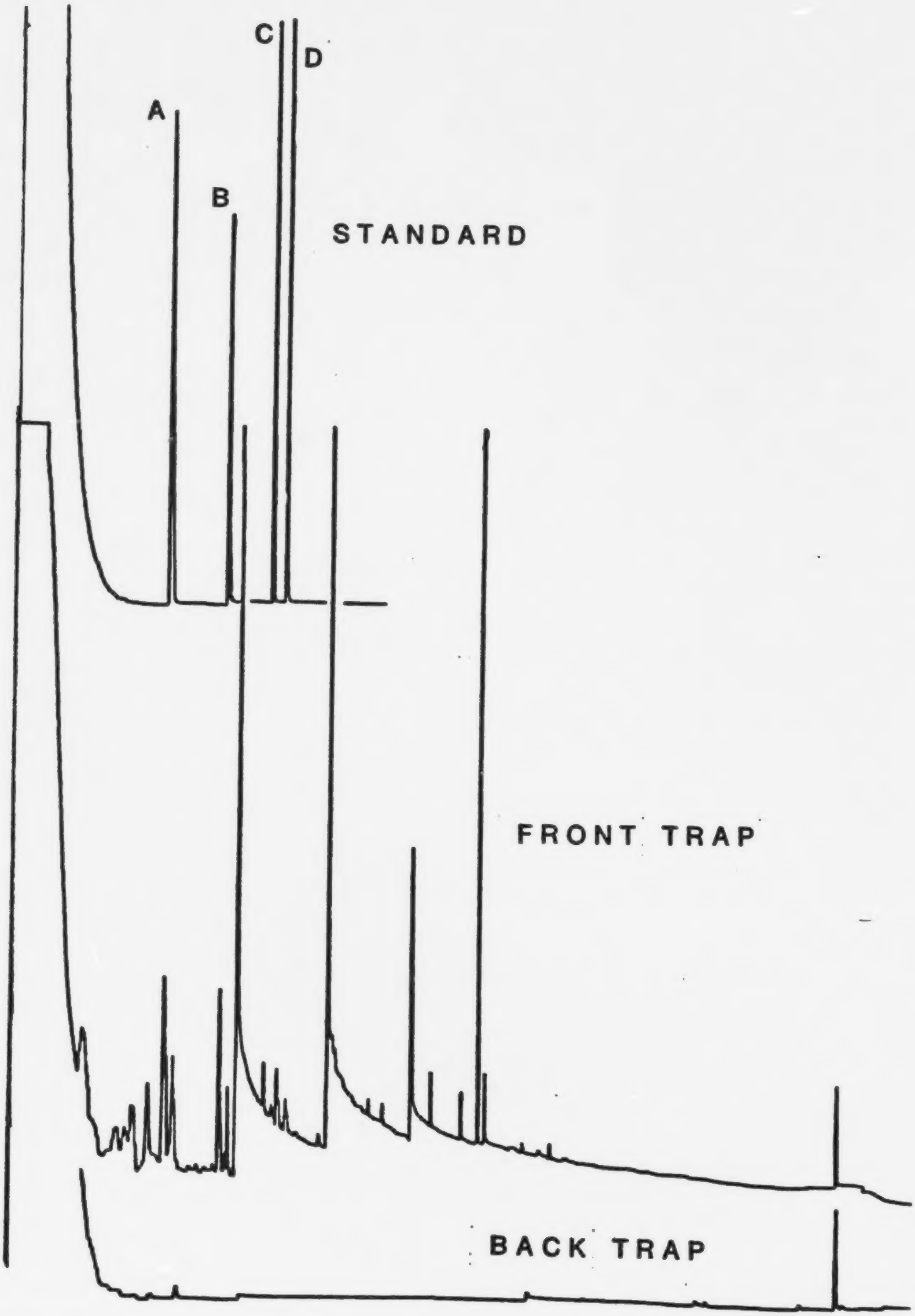


FIGURE 28. Ambient air sample 2 from Columbia, SC (December, 1985), fraction 1. Standard peaks are: A = m-DCB, B = p-DCB, C = o-DCB, D = 1,2,4-TCB, E = 1,2,4,5-TeCB, F = HCB. Peak G is PeCB that was not included in the standard, but which was later identified in the air sample. GC-ECD after sulfuric acid cleanup.

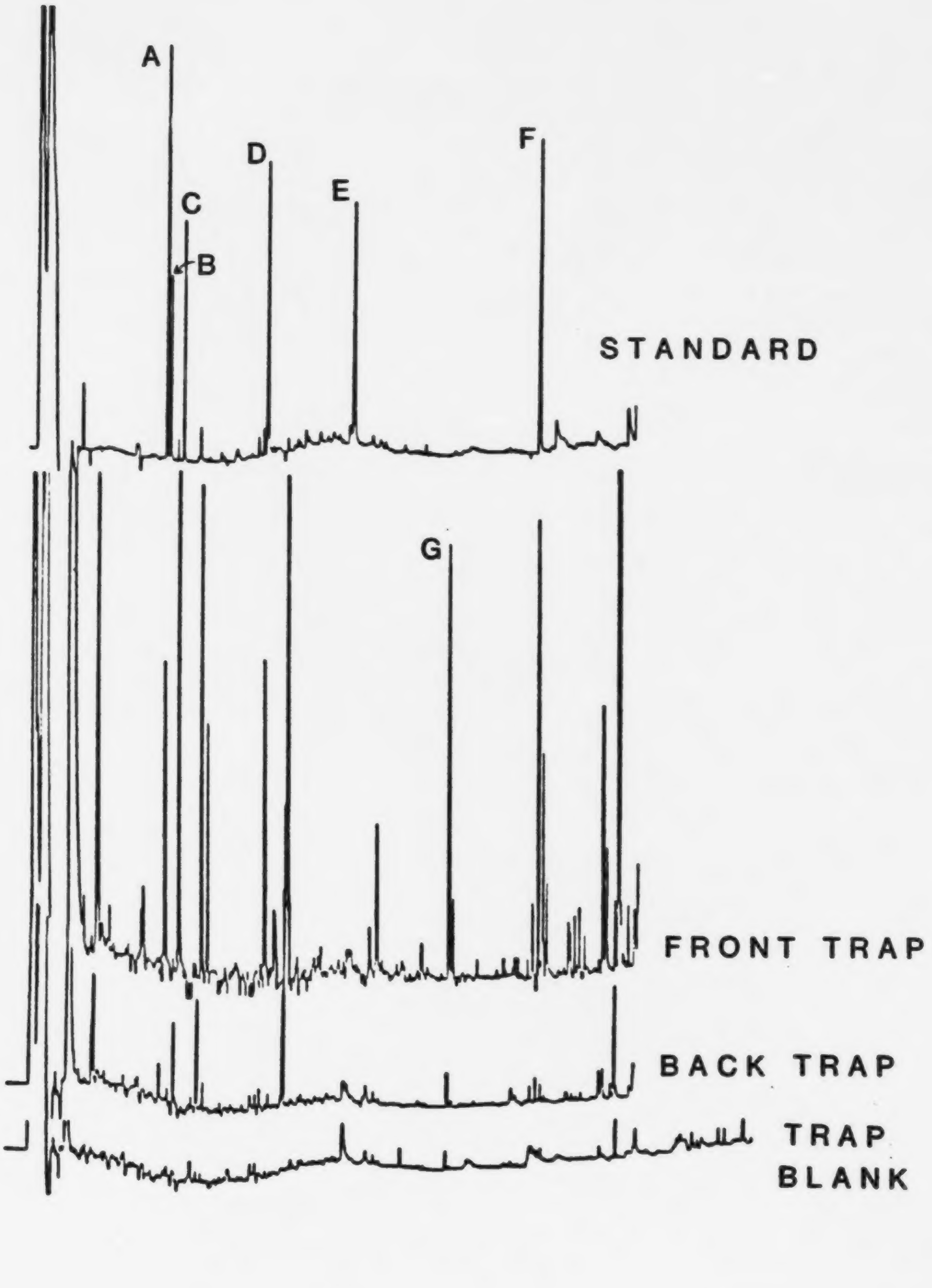


FIGURE 29. Reconstructed ion chromatograms (EI) of dichlorobenzenes in ambient air sample 2 (Columbia, December, 1985). Monitored ions were those in Table 35.

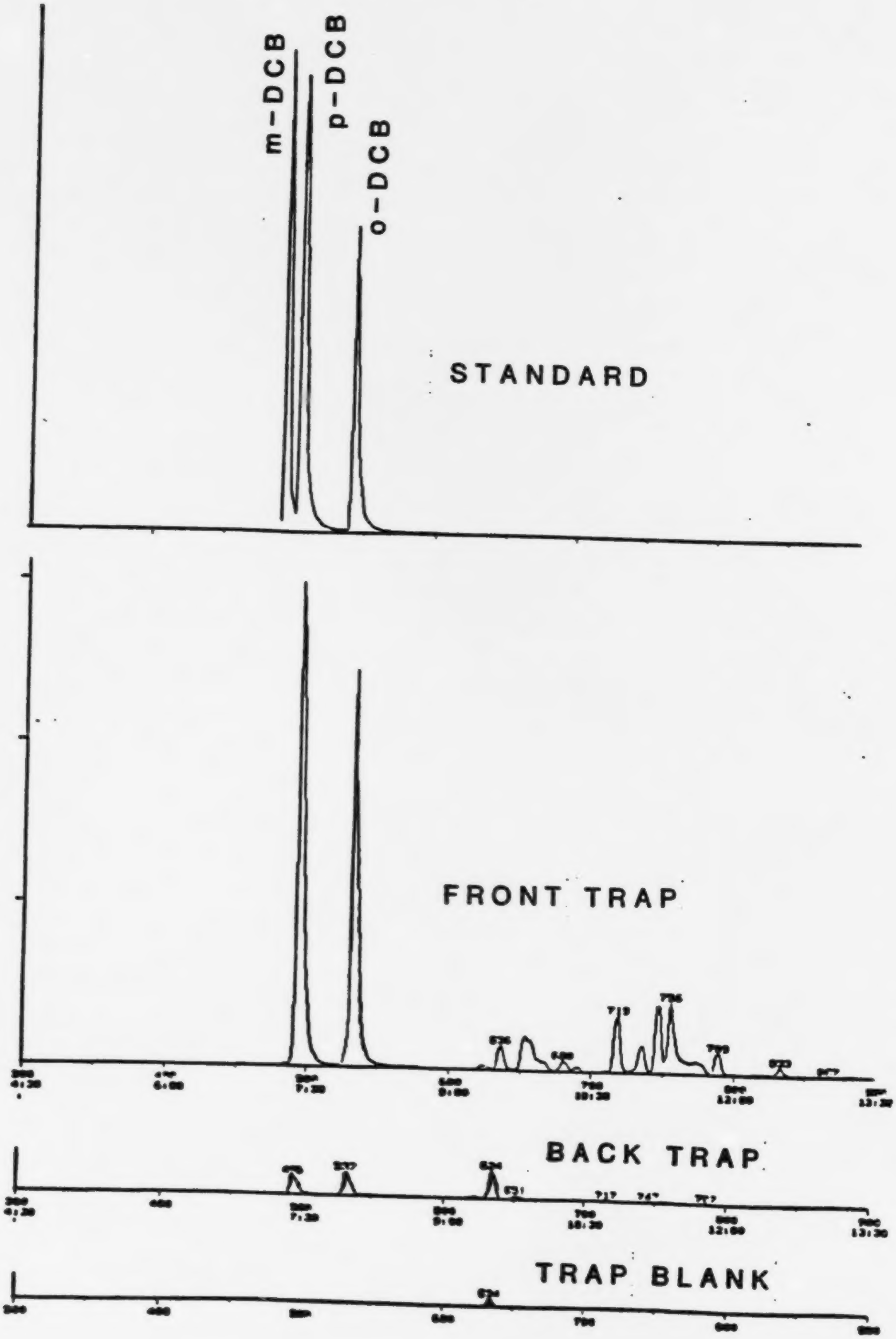


FIGURE 30. Reconstructed ion chromatograms (NI) of PeCB and HCB in ambient air sample 2 (Columbia, December, 1985). Monitored ions were those in Table 35.

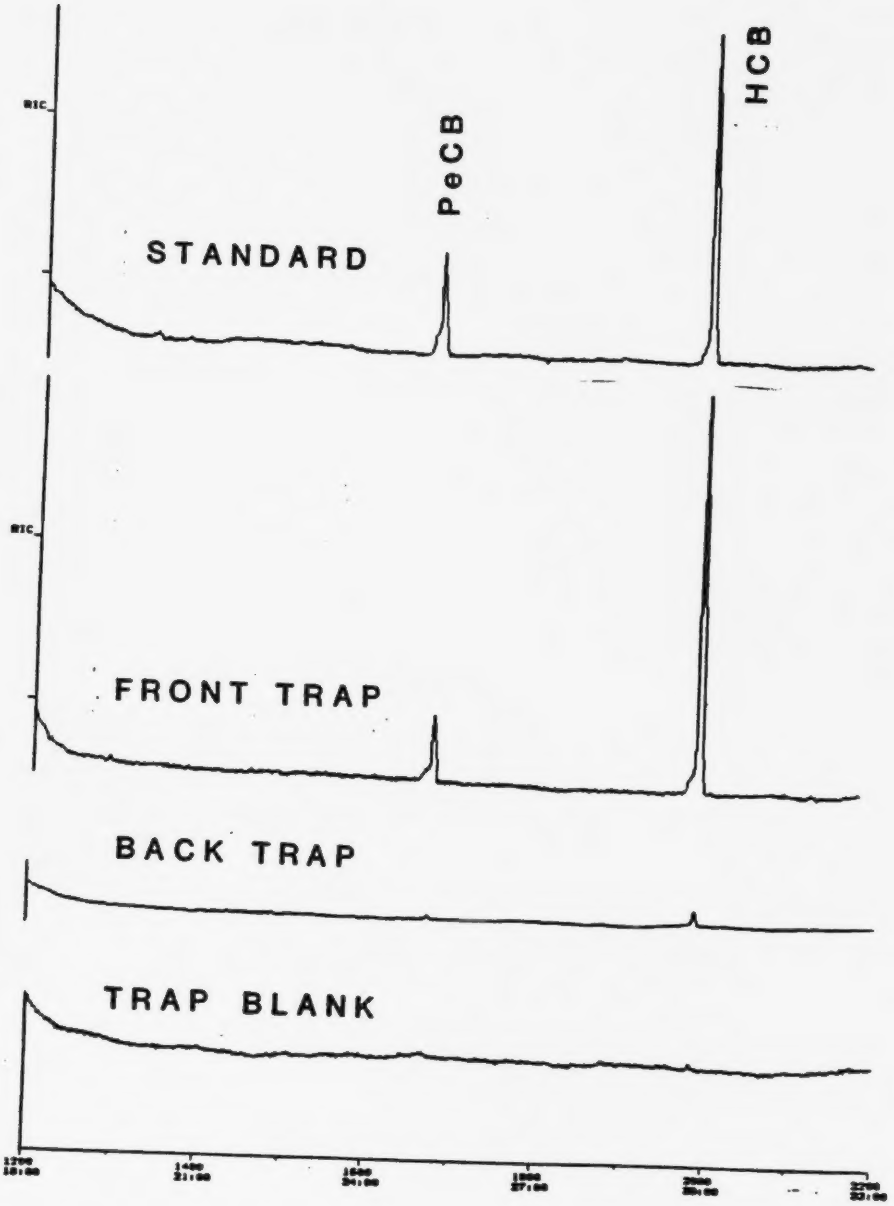


FIGURE 31. PCB in ambient air sample 2 (Columbia, December, 1985). GC-ECD after sulfuric acid cleanup.

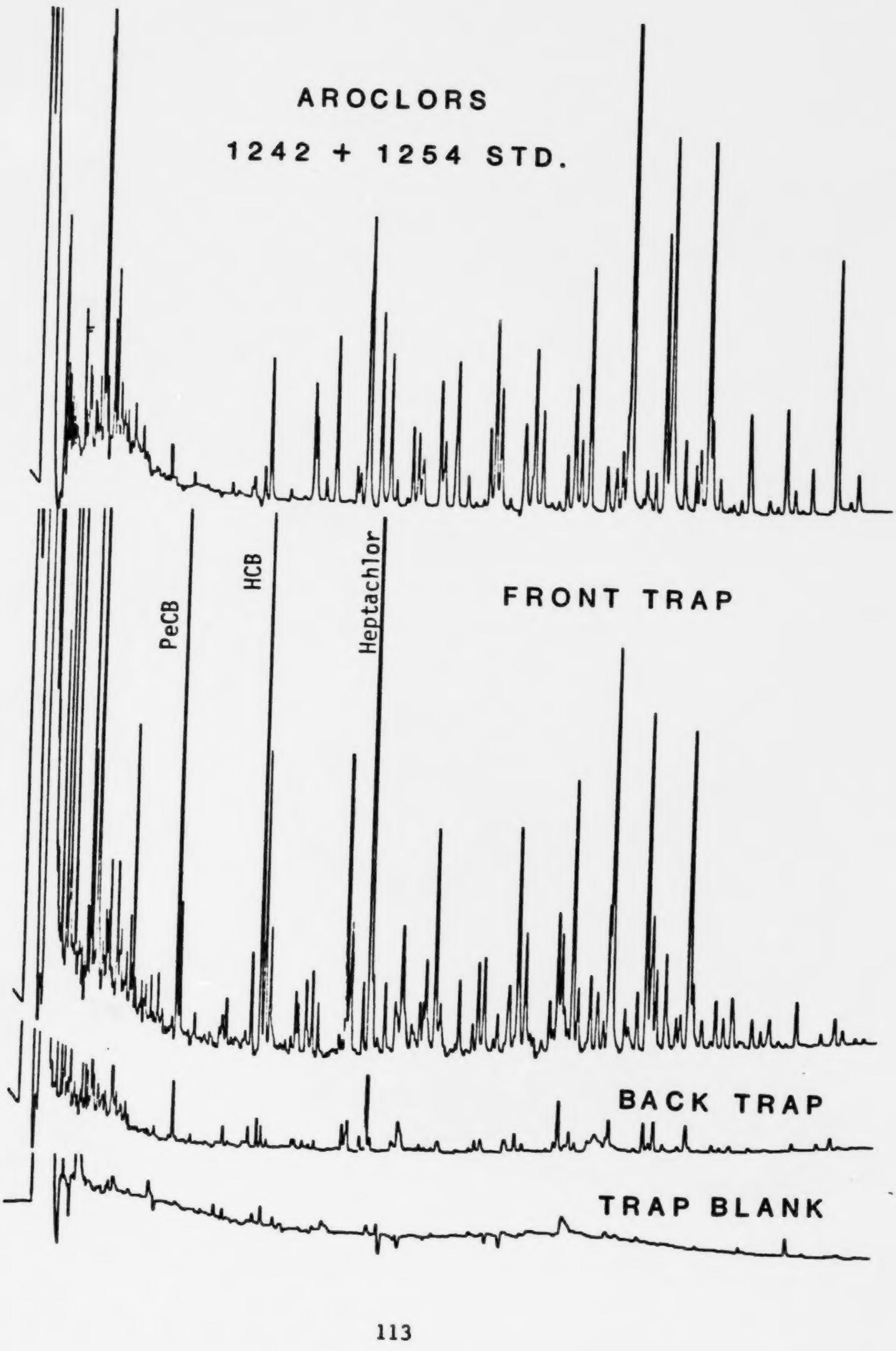


FIGURE 32. Organochlorine pesticides in ambient air sample 2 (Columbia, December, 1985). GC-ECD after sulfuric acid cleanup.

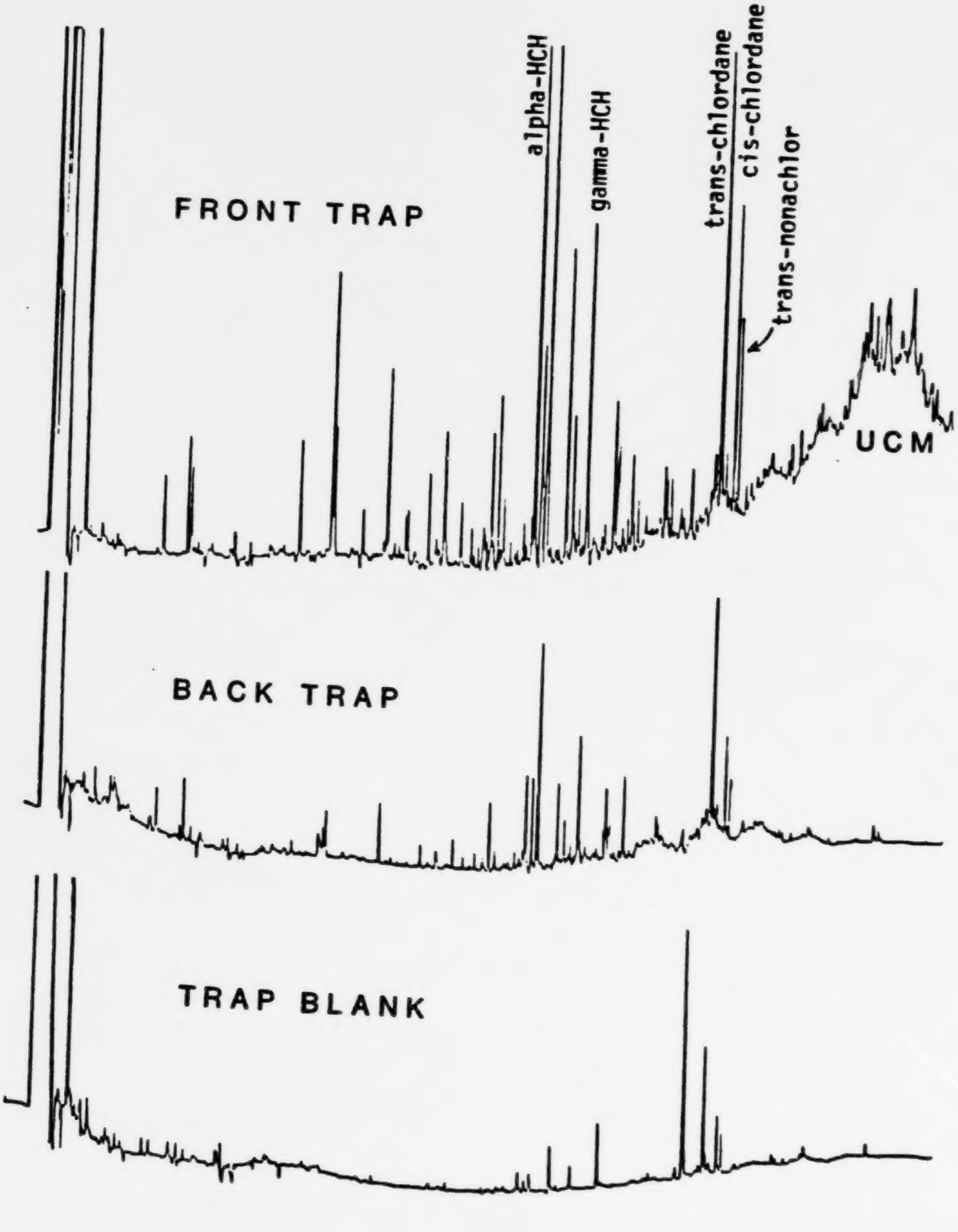


FIGURE 33. Ambient air sample 4, fraction 1, from North Inlet estuary, SC (November, 1986). Standard peaks are: A = m-DCB, B = p-DCB, C = o-DCB, D = 1,2,4-TCB, E = 1,2,4,5-TeCB, F = PeCB, G = α-HCH, H = HCB, I = γ-HCH. GC-ECD after sulfuric acid cleanup.

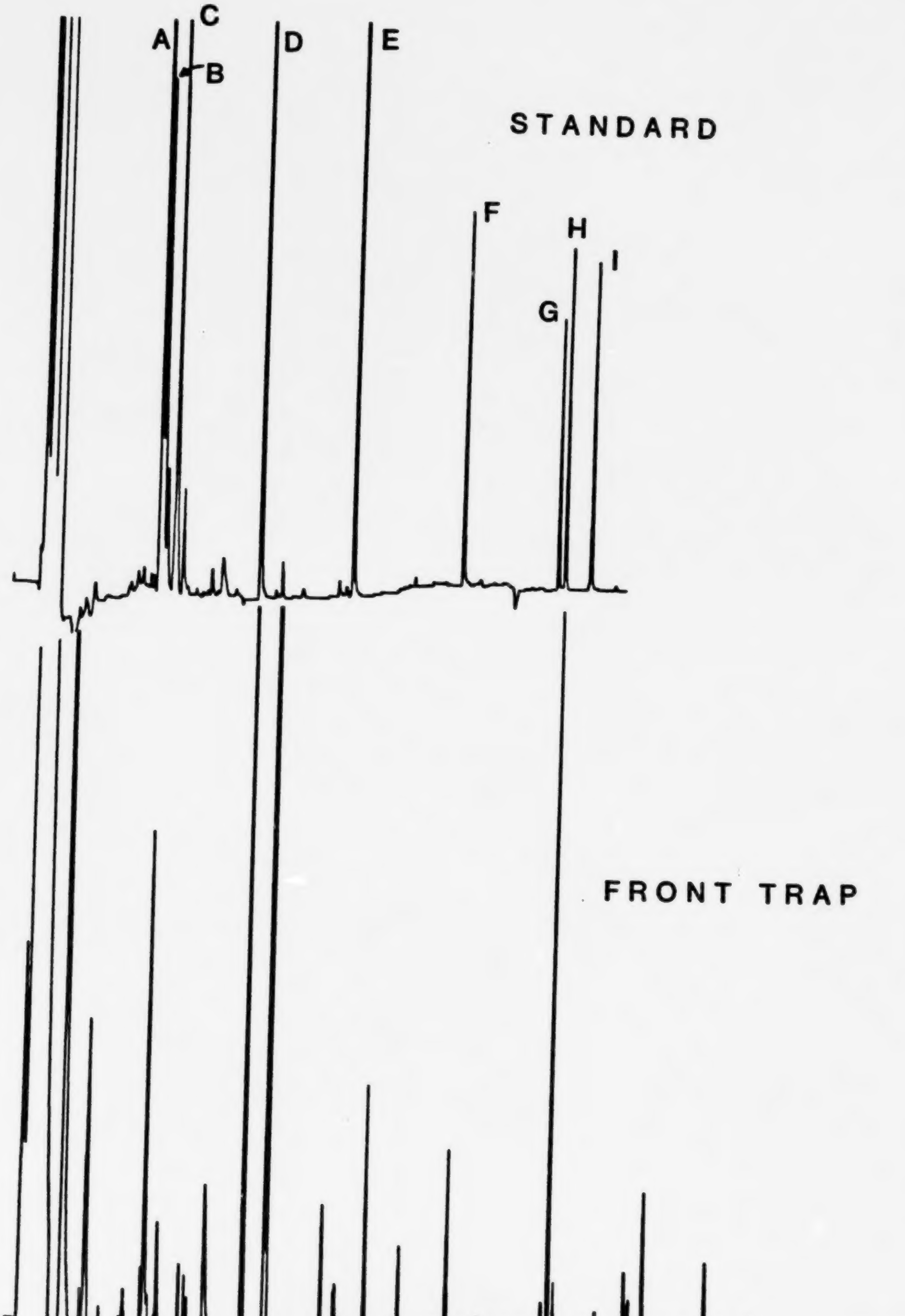


FIGURE 34. As in Figure 33, fraction 2. GC-ECD after sulfuric acid cleanup.

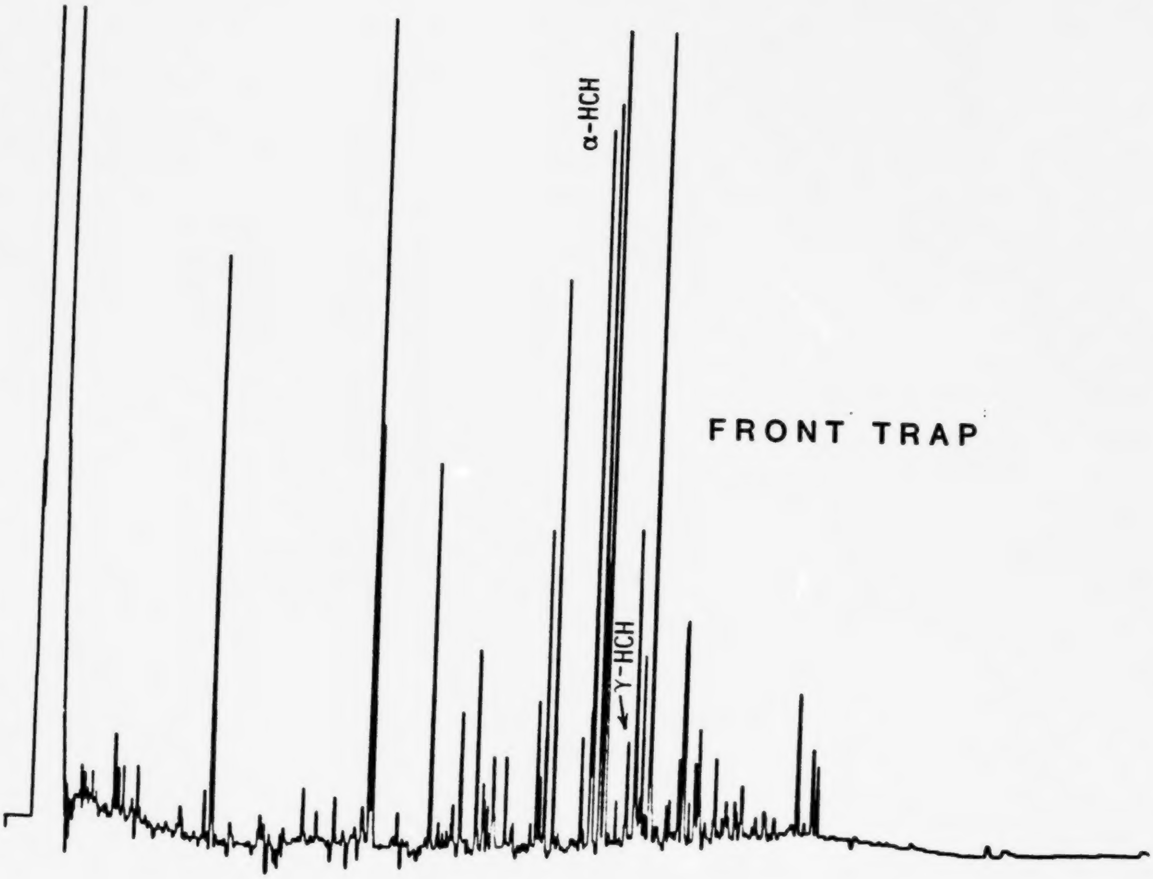


FIGURE 35. Ambient air sample 2 from Columbia, SC (December, 1985), chlorophenol fraction. Standard chlorophenol acetates are: A = 2,6-DCP, B = 2,4-DCP, C = 3,4-DCP, D = 2,4,6-TCP, E = 2,3,4,5-TeCP, F = PCP. GC-ECD.

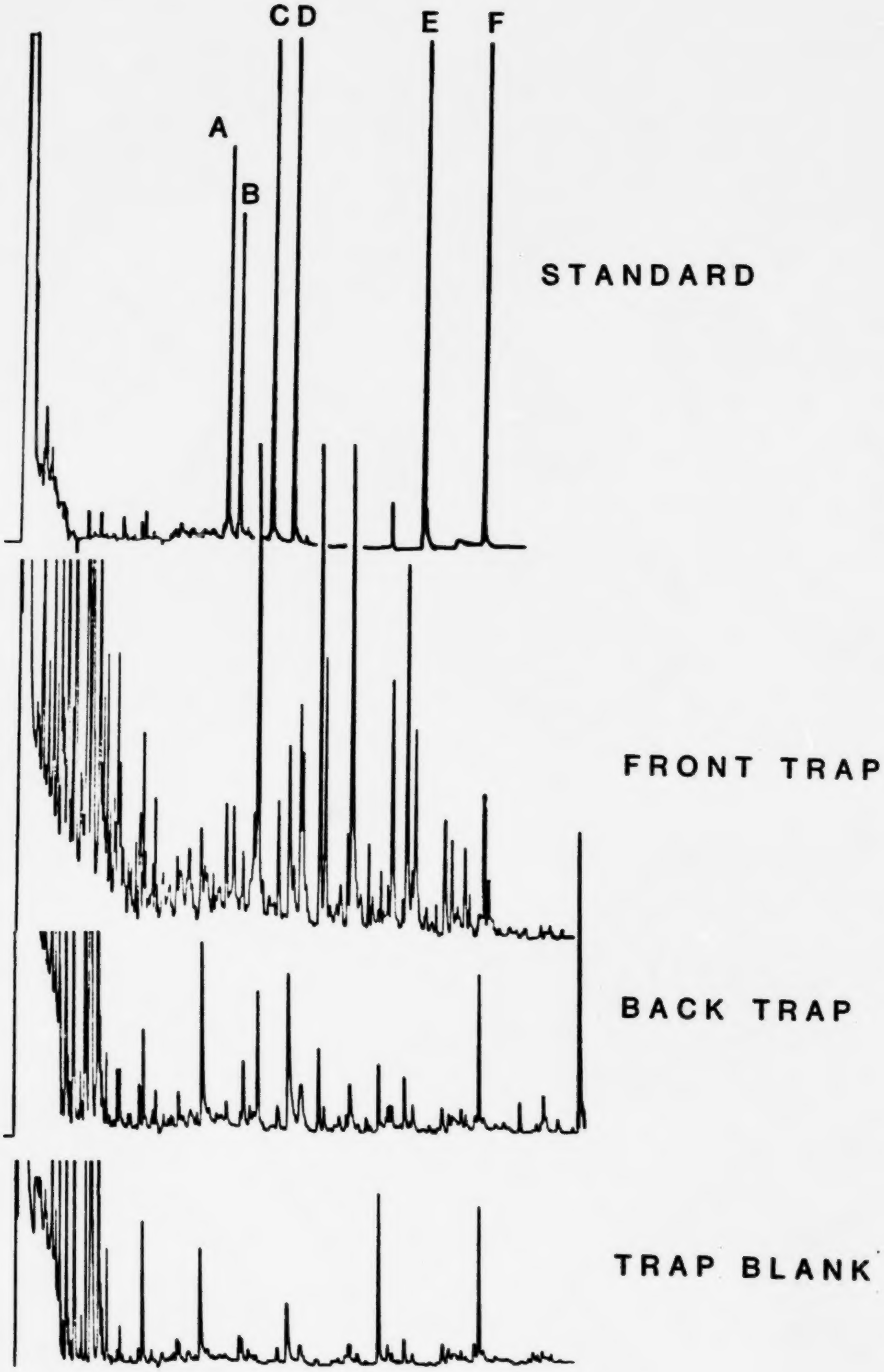
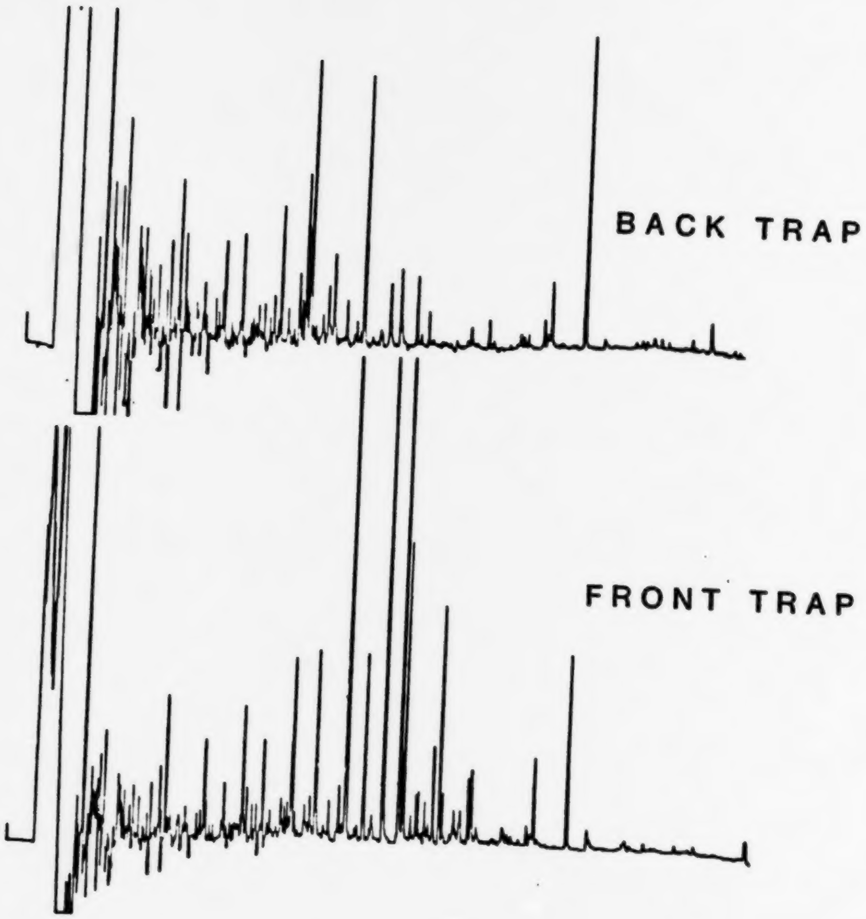


FIGURE 36. Ambient air sample 4 from North Inlet estuary, SC (November, 1986), chlorophenol fraction. GC-ECD.



SECTION 6

REFERENCES

Abrahamsson, K., Xie, T.M., Direct determination of trace amounts of chlorophenols in fresh water, waste water, and seawater. J. Chromatogr. 279, 199 (1983).

Atlas, E.L., Giam, C.S., Ambient concentrations and precipitation scavenging of atmospheric organic pollutants. Water, Air, Soil Pollut. (submitted).

Balson, E.W., Studies in vapor pressure measurement, part III. an effusion manometer sensitive to 5E-6 millimeters mercury: vapor pressures of DDT and other slightly volatile substances. Trans. Faraday Soc. 43, 54 (1947).

Bidleman, T.F., Estimation of vapor pressures for non-polar organic compounds by capillary gas chromatography. Anal. Chem. 56, 2490 (1984).

Bidleman, T.F., High volume collection of organic vapors using solid adsorbents. in: Lawrence, J.F. (ed.) Trace Analysis, Vol. 4, Academic Press, NY, pp. 51-100 (1985).

Bidleman, T.F., Simon, C.G., Burdick, N.F., You, F., Theoretical plate measurements and collection efficiencies for high-volume air samplers using polyurethane foam. J. Chromatogr. 301, 448 (1984).

Billings, W.N., Bidleman, T.F., Field comparison pf polyurethane foam and Tenax-GC resin for high volume air sampling of chlorinated hydrocarbons. Environ. Sci. Technol 14, 679 (1980).

Billings, W.N., Bidleman, T.F., High volume collection of chlorinated hydrocarbons in urban air using three solid adsorbents. Atmos. Environ. 17, 383 (1983).

Brown, R.H., Purnell, C.J., Collection and analysis of trace organic vapor pollutants in ambient atmospheres. The performance of a Tenax-GC tube. J. Chromatogr. 178, 79 (1979).

Burdick, N.F., Bidleman, T.F., Frontal movement of hexachlorobenzene and polychlorinated biphenyl vapors through polyurethane foam. Anal. Chem. 53, 1926 (1981).

Butler, L.D., Burke, M.F., Chromatographic characterization of porous polymers for use as adsorbents in sampling columns. J. Chromatogr. Sci. 14, 117 (1976).

Chau, A.S.Y., Coburn, J.A., Determination of pentachlorophenol in natural and waste waters. J. Assoc. Offic. Anal. Chem. 57, 389 (1974).

Clark, A.I., MacIntyre, J.N., Lester, J.N., Perry, J. Evaluation of a Tenax-GC sampling procedure for collection and analysis of vehicle-related aromatic and halogenated hydrocarbons in ambient air. J. Chromatogr. 252, 147 (1982).

Conder, J.R., Rees, G.J., McHale, S., Evaluation of methods of measuring gas-liquid chromatographic retention on skewed peaks. J. Chromatogr. 258, 1 (1983).

Conder, J.R., McHale, S., Jones, M.A., Evaluation of methods for measuring gas-solid chromatographic retention on skewed peaks. Anal. Chem. 58, 2663 (1986).

Doskey, P.V., Andren, A.W., Concentrations of airborne PCBs over Lake Michigan. J. Gt. Lakes Res. 7, 15 (1981).

Eiceman, G.A., Karasek, F.W., Retention behavior of chlorobenzenes, chlorophenols, aldrin, and Aroclor 1242 on Tenax-GC, Chromasorb 101, Florisil, and Carbopack C HT. J. Chromatogr. 200, 115 (1980).

Eisenreich, S.J., Looney, B.b., Hollod, G.J., PCBs in the Lake Superior atmosphere, 1978-80. in: Mackay, D., Paterson, S., Eisenreich, S.J., Simmons, M.S., Physical Behavior of PCBs in the Great Lakes, Ann Arbor Science, Ann Arbor, MI, pp. 115-125 (1983).

Farmer, C.T., Wade, T.L., Relationship of ambient atmospheric hydrocarbon (C-12 to C-32) concentrations to deposition. Water, Air, Soil Pollut. 29, 439 (1986).

Gallant, R.F., Characterization of sorbent resins for use in environmental sampling. EPA-600/7-78-054, U.S. Environmental Protection Agency, Research Triangle Park, NC (1978).

Harkov, R., Gianti, S.J., Jr., Bozzelli, J.W., LaRegina, J.E., Monitoring volatile organic compounds at hazardous and sanitary landfills in New Jersey. J. Environ. Sci. Health A20, 491 (1985).

Hanson, R.L., Clark, C.R., Carpenter, R.L., Hobbs, C.L., Evaluation of Tenax-GC and XAD-2 as polymer adsorbents for sampling fossil fuel combustion products containing nitrogen oxides. Environ. Sci. Technol. 15, 701 (1981).

Hunt, G.T., Pangaro, N., Potential contamination from the use of synthetic adsorbents in air sampling procedures. Anal. Chem. 54, 369 (1982).

Hunt, G.T., Ambient monitoring of semivolatile organics using high volume sorbent samplers -- critical quality control features and other considerations in sample collection and network design. Presented at the EPA/APCA Symposium on Measurement of Toxic Air Pollutants, Raleigh, NC, April (1986).

Keller, C.D., Bidleman, T.F., Collection of airborne polycyclic aromatic hydrocarbons and other organics with a glass fiber filter - polyurethane foam system. Atmos. Environ. 18, 837 (1984).

Krost, K.J., Pellizzari, E.D., Walburn, S.G., Hubbard, S.A., Collection and analysis of hazardous organic emissions. Anal. Chem. 54, 810 (1982).

Lewis, R.G., Jackson, M.D., Modification and evaluation of a high volume air sampler for pesticides and semivolatile industrial organic chemicals. Anal. Chem. 54, 592 (1982).

Lewis, R.G., MacLeod, K.E., Portable sampler for pesticides and semivolatile industrial organic chemicals. Anal. Chem. 54, 310 (1982).

Ligocki, M.P., Leuenberger, C., Pankow, J.F., Trace organic compounds in rain. II. gas scavenging of neutral organic compounds. Atmos. Environ. 19, 1609 (1985).

Mackay, D., Bobra, A., Chan, D.W., Shiu, W.-Y., Vapor pressure correlations for low volatility environmental chemicals. Environ. Sci. Technol. 16, 645 (1982).

Murphy, T.J., Rzeszutko, C.P., Polychlorinated biphenyls in precipitation in the Lake Michigan basin. EPA-600/3-78-071, U.S. Environmental Protection Agency, Grosse Ile, MI (1978).

Neher, M.B., Jones, D.W., In-situ decomposition product isolated from Tenax-GC while sampling stack gases. Anal. Chem. 49, 572 (1977).

Oehme, M., Ottar, B., The long range transport of polychlorinated hydrocarbons to the Arctic. Geophys. Res. Lett. 11, 1133 (1984).

Pellizzari, E.D., Demian, B., Krost, K., Sampling organic compounds in the presence of reactive inorganic gases with Tenax-GC. Anal. Chem. 56, 793 (1984).

Pellizzari, E.D., Analysis of organic air pollutants by gas chromatography and mass spectrometry. EPA-600/2-77-100, U.S. Environmental Protection Agency, Research Triangle Park, NC (1977).

Reilley, C.N., Hildebrand, G.P., Ashley, J.W., Gas chromatographic response as a function of sample input profile. Anal. Chem. 34, 1198 (1962).

Senum, G., Theoretical collection efficiencies for adsorbent samplers. Environ. Sci. Technol. 15, 1073 (1981).

Simon, C.G., Bidleman, T.F., Sampling airborne PCB with polyurethane foam: a chromatographic approach to determining retention efficiencies. Anal. Chem. 51, 1110 (1979).

Wallace, L., Pellizzari, E.D., Hartwell, T.D., Sparacino, C.M., Sheldon, L.S., Zelon, H., Personal exposures, indoor-outdoor relationships, and breath levels of toxic air pollutants measured for 355 persons in New Jersey. Atmos. Environ. 19, 1651 (1985).

Weast, R.C., Handbook of Chemistry and Physics, , 52nd ed., CRC Press, pp. D151 - D170 (1971).

Van Der Straeten, D., Langenhove, H., Schamp, N., Comparison between theoretical and experimental sampling efficiencies on Tenax. J. Chromatogr. 331, 207 (1985).

Van Vaeck, L., Van Cauwenberghe, K., Janssens, J., The gas-particle distribution of organic aerosol constituents: measurement of the volatilization artifact in hi-vol cascade impactor sampling. Atmos. Environ. 18, 417 (1984).

West, D.S., Hodgson, F.N., Brooks, J.J., DeAngelis, G., Potential atmospheric carcinogens: phase 2/3, analytical techniques and field evaluation. EPA-600/2-81-106, U.S. Environmental Protection Agency, Research Triangle Park, NC (1981).

SECTION 7

APPENDIX. DATA FOR INDIVIDUAL COLLECTION EFFICIENCY EXPERIMENTS AND COMPARISON SPREADSHEETS

TABLE A1

Collection Experiment #1 Tenax in Stacked Sieves.

Collection Data

Time: 300 min Flow: 0.33m^3/min Total Volume: 100m^3 Temp: 20°C

Trap 1: 3.9 g Tenax

Trap 2: 3.9 g Tenax

Trap 3: 3.9 g Tenax

Trap 4: 2.0 g Tenax

Compound	µg Injected	µg Found[a] T-1	T-2	T-3	T-4	Total	% of injected
p-DCB	0	3.14	4.27	0.73	2.67	10.81	-
o-DCB	0	0.64	0.05	0.05	0.05	0.79	-
1,2,4-TCB	0	0.12	0.36	0.42	0.62	1.52	-
Hexa-CB	0	0.029	0.001	0.005	0.014	0.049	-
NAP	752	528	150	17	<8	695	92.4
2-MN	440	362	65	<8	<8	427	97.1
BIPH	508	454	19	<8	<8	473	93.1
2,6-DMN	593	519	42	<8	<8	561	94.6

---

a) uncorrected for procedural recovery, but corrected for blanks

TABLE A2

Collection Experiment #2 Tenax Traps in Stacked Sieves.

Collection Data

Time: 300 min Flow:0.33m^3/min Total Volume: 100m^3 Temp: 20°C

Trap 1: 3.9 g Tenax

Trap 2: 3.9 g Tenax

Trap 3: 3.9 g Tenax

Trap 4: 3.9 g Tenax

Compound	µg Injected	µg Found[a] T-1	T-2	T-3	T-4	Total	% of injected
o-DCB	16.11	3.49	2.80	1.32	1.22	8.83	54.8
1,2,4-TCB	10.83	6.40	0.88	ND	ND	7.28	67.2
Hexa-CB	0.482	0.396	0.013	0.004	0.004	0.416	93.4
NAP	0	0.25	ND	ND	ND	-	-
2-MN	0	0.26	ND	ND	ND	-	-
BIPH	0	1.98	ND	ND	ND	-	-
2,6-DMN	0	0.19	ND	ND	ND	-	-
TEO[b]	0	94	17	13	17	141	-

ND = not determined

a) uncorrected for procedural recovery, but corrected for blanks

b) TEO = total extractable organics eluting from 5-23 min, and quantified by GC-FID

TABLE A3

Collection Experiment #3 PUF-Tenax Sandwich in PS-1 Sampler.

Collection Data

Time: 258 min Flow:0.14m^3/min Total Volume:33.6m^3 Temp: 20°C

Trap 1: 2 cm PUF	Trap 3: 2 cm PUF	Trap 5: 2 cm PUF
Trap 2: 10 g Tenax	Trap 4: 3 g Tenax	

Compound	µg Injected	µg Found[a] T-1	T-2	T-3	T-4	T-5	Total	% of injected
p-DCB	20.9	0.2	17.0	-	0.6	-	17.8	85.1
o-DCB	43.2	0.5	32.3	-	<0.1	-	32.8	75.9
1,2,4-TCB	14.7	<0.1	15.8	-	<0.1	-	15.8	107.5
Hexa-CB	0.656	0.173	0.648	-	<0.02	-	0.821	125.1
NAP	721	-	567	-	2	-	569	78.9
2-MN	422	-	347	-	<2	-	347	82.2
BIPH	487	-	402	-	<2	-	402	82.5
2,6-DMN	569	-	474	-	<2	-	474	83.3

---

a) uncorrected for procedural recovery, but corrected for blanks

TABLE A4

Collection Experiment #4 PUF-Tenax Sandwich in PS-1 Sampler.

Collection Data

Time: 245 min Flow:0.14m^3/min Total Volume: 34 m^3 Temp: 20°C

Trap 1: 2 cm PUF	Trap 3: 2 cm PUF	Trap 5: 2 cm PUF
Trap 2: 10 g Tenax	Trap 4: 3.5 g Tenax	

Compound	µg Injected		µg Found[a]					% of injected
		T-1	T-2	T-3	T-4	T-5	Total	
p-DCB	0	ND	0.37	-	0.6	-	0.97	-
o-DCB	0	0.11	0.22	-	<0.1	-	0.33	-
1,2,4-TCB	0	ND	0.22	-	<0.1	-	0.22	-
Hexa-CB	0	0.002	<0.001	<0.001	<0.02	-	0.002	-
NAP	391	ND	371	5	2	-	377	96.4
2-MN	229	ND	223	-	<2	-	223	97.2
BIPH	264	2	257	-	<2	-	259	98.1
2,6-DMN	308	1	303	-	<2	-	304	98.3

---

a) uncorrected for procedural recovery, but corrected for blanks

TABLE A5

Collection Experiment #5 PUF-Tenax Sandwich in PS-1 Sampler.

Collection Data

Time: 230 min Flow:0.14m^3/min Total Volume:32.2m^3 Temp: 20°C

Trap 1: 2 cm PUF	Trap 3: 2 cm PUF	Trap 5: 2 cm PUF
Trap 2: 10 g Tenax	Trap 4: 3.2 g Tenax	

Compound	μg Injected	T-1	μg Found[a] T-2	T-3	T-4	T-5	Total	% of injected
p-DCB	19.6	ND	16.5	ND	0.6	ND	17.1	87.2
o-DCB	20.6	ND	17.4	ND	0.8	ND	18.2	88.3
1,2,4-TCB	3.47	ND	3.69	ND	0.01	ND	3.70	106.6
Hexa-CB	0.154	ND	0.117	ND	0.002	ND	0.119	77.2
NAP	0	ND	ND	ND	ND	ND	-	-
2-MN	0	ND	ND	ND	ND	ND	-	-
BIPH	0	ND	ND	ND	ND	ND	-	-
2,6-DMN	0	ND	ND	ND	ND	ND	-	-

ND = not determined

a) uncorrected for procedural recovery, but corrected for blanks

TABLE A6

Collection Experiment #6 PUF-Tenax Sandwich in PS-1 Sampler.

Collection Data

Time: 648 min Flow: $0.18 m^3/min$ Total Volume: $119 m^3$ Temp: 20°C

Trap 1: 2 cm PUF

Trap 2: 9.9 g Tenax

Trap 3: 2 cm PUF

Trap 4: 3.2 g Tenax

Trap 5: 2 cm PUF

Compound	µg Injected	T-1	µg Found[a] T-2	T-3	T-4	T-5	Total	% of injected
p-DCB	0	0.02	0.32	0.04	0.09	ND	0.47	-
o-DCB	0	0.14	ND	0.04	ND	ND	0.18	-
1,2,4-TCB	0	0.007	ND	ND	ND	ND	0.007	-
Hexa-CB	0	0.006	<0.001	<0.001	ND	ND	0.006	-
NAP	308	3	276	ND	2	ND	281	91.2
2-MN	180	4	163	ND	ND	ND	167	92.8
BIPH	208	10	186	ND	ND	ND	196	94.2
2,6-DMN	243	8	222	ND	ND	ND	230	94.7

ND = not determined

a) uncorrected for procedural recovery, but corrected for blanks

TABLE A7

Collection Experiment #7 PUF-Tenax Sandwich in PS-1 Sampler.

Collection Data

Time: 660 min Flow: 0.18 m^3/min Total Volume: 117 m^3 Temp: 20°C

Trap 1: 2 cm PUF Trap 3: 2 cm PUF Trap 5: 2 cm PUF

Trap 2: 9.7 g Tenax Trap 4: 3.3 g Tenax

Compound	μg Injected	μg Found[a] T-1	T-2	T-3	T-4	T-5	Total	% of injected
p-DCB	15.3	ND	10.2	0.4	0.7	ND	11.3	73.9
o-DCB	14.5	ND	9.9	<0.1	1.1	ND	11.1	76.6
1,2,4-TCB	2.56	ND	2.4	<0.1	0.02	ND	2.42	94.5
Hexa-CB	0.114	ND	0.112	<0.001	<0.001	ND	0.112	98.2
NAP[b]	269	6	425	4	36	ND	471	175
2-MN	158	1	145	ND	ND	ND	146	92.4
BIPH	183	9	169	ND	ND	ND	178	97.3
2,6-DMN	213	7	202	ND	ND	ND	209	98.1

ND = not determined

a) uncorrected for procedural recovery, but corrected for blanks

b) contamination from new XAD-2 prefilter

TABLE A8

Collection Experiment #8 PUF-Tenax Sandwich in PS-1 Sampler.

Collection Data

Time: 900 min Flow: 0.18 m^3/min Total Volume: 165 m^3 Temp: 20°C

Trap 1: 2 cm PUF
Trap 2: 9.6 g Tenax
Trap 3: 2 cm PUF
Trap 4: 3.1 g Tenax
Trap 5: 2 cm PUF

Compound	µg Injected	µg Found[a]						% of injected
		T-1	T-2	T-3	T-4	T-5	Total	
p-DCB	17.4	<0.1	11.7	0.1	1.4	ND	13.2	75.9
o-DCB	18.3	0.2	13.7	0.1	1.7	ND	15.7	86.3
1,2,4-TCB	3.08	0.05	3.4	<0.01	0.03	ND	3.48	113
Hexa-CB	0.137	0.008	0.129	0.001	0.001	ND	0.139	101
NAP	259	2	197	0.5	5	ND	204	78.8
2-MN	152	3	120	ND	ND	ND	123	80.9
BIPH	175	8	132	ND	ND	ND	140	80.0
2,6-DMN	204	7	158	ND	ND	ND	165	80.9

ND = not determined

a) uncorrected for procedural recovery, but corrected for blanks

TABLE A9

Collection Experiment #9 PUF-Tenax Sandwich in PS-1 Sampler.

Collection Data

Time: 432 min Flow:0.18m^3/min Total Volume:77.8m^3 Temp: 20°C

Trap 1: 2 cm PUF	Trap 3: 2 cm PUF	Trap 5: 2 cm PUF
Trap 2: 10 g Tenax	Trap 4: 3 g Tenax	

Compound	µg Injected	µg Found[a] T-1	T-2	T-3	T-4	T-5	Total	% of injected
p-DCB	2.01	ND	1.08	ND	<0.01	ND	1.08	53.8
o-DCB	1.92	0.19	1.51	ND	0.11	ND	1.81	94.3
1,2,4-TCB	.34	0.016	.36	ND	ND	ND	.376	111
Hexa-CB	0.015	<0.006	0.016	ND	ND	ND	0.016	106
NAP	29.7	0.98	24.8	0.06	0.32	ND	26.2	88.1
2-MN	17.4	1.12	12.9	ND	0.08	ND	14.7	81.2
BIPH	20.1	1.99	13.7	ND	ND	ND	15.7	78.3
2,6-DMN	23.4	2.04	16.2	0.02	0.03	ND	18.3	78.1

ND = not determined

a) uncorrected for procedural recovery, but corrected for blanks

TABLE A10

Collection Experiment #10 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 468min Flow:0.22m^3/min Total Volume: 102m^3 Temp: 20^{o}C

Trap 1: PUF/ 10 g Tenax/ PUF

Trap 2: 3 g Tenax/ PUF

Compound	µg Injected	µg Found Trap 1	 Trap 2	% of injected Trap 1	 Trap 2
p-DCB	2.06	1.49	0.09	72	4
o-DCB	1.96	1.48	0.10	76	5
1,2,4-TCB	0.35	0.29	0.043	83	12
Hexa-CB	0.015	0.012	ND	80	-
NAP	29.7	21.4	0.69	72	2
2-MN	17.4	13.2	0.09	76	0.5
BIPH	20.1	17.0	0.07	85	0.3
2,6-DMN	23.4	20.4	0.20	87	0.9

ND = not determined

TABLE A11

Collection Experiment #11 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 570min Flow:0.22m^3/min Total Volume: 125m^3 Temp: 20°C

Trap 1: PUF/ 10 g Tenax/ PUF

Trap 2: 3 g Tenax/ PUF

Compound	μg Injected	μg Found Trap 1	μg Found Trap 2	% of injected Trap 1	% of injected Trap 2
p-DCB	2.04	1.21	0.10	59	5
o-DCB	1.94	1.21	0.17	62	9
1,2,4-TCB	0.34	0.31	0.002	91	0.6
Hexa-CB	0.015	0.014	ND	93	-
NAP	29.0	22.1	ND	76	2
2-MN	17.0	14.1	0.27	83	2
BIPH	19.6	15.6	ND	80	-
2,6-DMN	22.9	18.7	0.15	82	0.7

ND = not determined

TABLE A12

Collection Experiment #12 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 444min Flow:0.22m^3/min Total Volume: 125m^3 Temp: 20°C

Trap 1: PUF/ 10 g Tenax/ PUF

Trap 2: 3 g Tenax/ PUF

Compound	μg Injected	μg Found Trap 1	 Trap 2	% of injected Trap 1	 Trap 2
p-DCB	0.0	ND	0.013	-	-
o-DCB	0.0	0.08	0.018	-	-
1,2,4-TCB	0.0	0.011	0.006	-	-
Hexa-CB	0.0	0.0002	0.0006	-	-
NAP	0.0	ND	ND	-	-
2-MN	0.0	ND	ND	-	-
BIPH	0.0	ND	0.393	-	-
2,6-DMN	0.0	ND	0.023	-	-
TEO[a]	0.0	47.85	147.9	-	-

ND = not determined

a) Total extractable organics eluting 3-16 min, and quantified by GC-FID

TABLE A13

Collection Experiment #13 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1440 min Flow:0.257m^3/min Total Volume: 346 m^3 Temp: 20°C

Prefilter: charcoal

Trap 1: PUF/ 10g Tenax/ PUF

Trap 2: 3g Tenax/ PUF

Compound	ng Injected	ng Found[a] Trap 1	 Trap 2	% of injected Trap 1	 Trap 2
Organochlorines:					
m-DCB	2026	<544	<400	<27	<20
p-DCB	2143	<1007	<860	<47	<40
o-DCB	1513	<520	<460	<35	<30
1,2,4-TCB	320	<250	<80	<78	<25
1,2,4,5-TeCB	198	173	<80	89	<40
PentaCB	21	19.6	<5	93	<24
HexaCB	13	12.7	<5	98	<38
alpha-HCH	13	8.8	<5	68	<38
gamma-HCH	17	19.1	<6	112	<35
Chlorophenols:					
2,6-DCP	370	392	<80	106	<22
2,4-DCP	346	515	<100	149	<29
3,4-DCP	699	780	<80	116	<11
2,4,6-TCP	170	180	<3	106	<2
2,3,4,5-TeCP	77	49	<8	65	<10
Penta-CP	43	15	13	35	30

---

a) corrected for average operational blank in case of organochlorines, but not in case of chlorophenols

TABLE A14

Collection Experiment #14 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1440 min Flow:0.257m^3/min Total Volume: 358 m^3 Temp: 20°C

Prefilter: Tenax

Trap 1: 5 g Tenax between 2 PUF slices.

Trap 2: 5 g Tenax between 2 PUF slices.

Compound	ng Injected	ng Found[a]		% of injected	
		Trap 1	Trap 2	Trap 1	Trap 2
Organochlorines:					
m-DCB	3200	<500	<500	<16	<16
p-DCB	3400	<1000	3529	<29	104
o-DCB	2400	<600	3720	<25	155
1,2,4-TCB	510	522	<200	102	<39
1,2,4,5-TeCB	310	323	<90	104	<29
PentaCB	34	34.6	<5	102	<15
HCB	20	21.7	<5	109	<25
alpha-HCH	20	15.8	<5	79	<25
gamma-HCH	28	23	<6	83	<21
Chlorophenols:					
2,6-DCP	540	430	86	79	15
2,4-DCP	510	500	58	98	11
3,4-DCP	1000	850	<40	85	<4
2,4,6-TCP	240	220	<20	90	<8
2,3,4,5-TeCP	110	71	3	63	3
Penta-CP	64	11	570	18	900?

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a) corrected for average operational blank in case of organochlorines, but not in case of chlorophenols

TABLE A15

Collection Experiment #15 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1440 min Flow:0.257m^3/min Total Volume: 353 m^3 Temp:20°C

Prefilter: charcoal

Trap 1: 5 g Tenax between 2 PUF slices ("old" Tenax from C.Sparacino.)

Trap 2: 5 g Tenax between 2 PUF slices " " " "

Compound	ng Injected	ng Found[a] Trap 1	Trap 2	% of injected Trap 1	Trap 2
Organochlorines:.					
m-DCB	3200	<420	<420	<13	<13
p-DCB	3400	<830	<830	<24	<24
o-DCB	2400	<440	<440	<18	<18
1,2,4-TCB	500	202	<95	40	<19
1,2,4,5-TeCB	310	273	<78	88	<25
PentaCB	33	32.6	<5	99	<15
Hexa-CB	20	18.7	<5	94	<25
alpha-HCH	18	11.8	<5	66	<28
gamma-HCH	27	23.1	<6	86	<22
Chlorophenols:					
2,6-DCP	510	480	55	94	11
2,4-DCP	480	560	<51	118	<11
3,4-DCP	970	970	<40	100	<4
2,4,6-TCP	230	220	<10	95	<4
2,3,4,5-TeCP	110	90	<3	85	<3
Penta-CP	60	20	3	34	5

---

a) corrected for average operational blank in case of organochlorines, but not in case of chlorophenols

TABLE A16

Collection Experiment #16 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1443min Flow:0.257m^3/min Total Volume: 372m^3 Temp: 20-22°C

Prefilter: charcoal

Trap 1: PUF/ 14.3 g XAD-2/ PUF

Trap 2: 14.3 g XAD-2/ PUF

Compound	ng Injected	ng Found		% of injected	
		Trap 1	Trap 2	Trap 1	Trap 2
Organochlorines:					
m-DCB	3146	<139	<135	<5	<5
p-DCB	3327	754	9500	12	275
o-DCB	2348	<160	<155	<7	<7
1,2,4-TCB	492	378	270	<76	<55
1,2,4,5-TeCB	307	362	43	118	14
PentaCB	33	49.0	5.9	149	18
Hexa-CB	20	26.3	<4.1	132	<21
alpha-HCH	20	20.2	<11.7	102	<59
gamma-HCH	27	29.2	<6.1	109	<23
Chlorophenols:					
2,6-DCP	606	431	48	71	8
2,4-DCP	566	436	71	77	13
3,4-DCP	1146	857	<30	75	<3
2,4,6-TCP	270	186	<4.4	69	<2
2,3,4,5-TeCP	125	44	<2.1	35	<2
Penta-CP	71	14	5.3	19	7.5

TABLE A17

Collection Experiment #17 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1444 min Flow:.257m^3/min Total Volume: 372m^3 Temp: 22-23°C

Prefilter: charcoal

Trap 1: PUF/ 14.3 g XAD-2/ PUF

Trap 2: 13.7 g XAD-2/ PUF

Compound	ng Injected	ng Found Trap 1	ng Found Trap 2	% of injected Trap 1	% of injected Trap 2
Organochlorines:					
m-DCB	4084	566	237	14	6
p-DCB	4319	2261	1442	52	33
o-DCB	3049	4999	840	164	28
1,2,4-TCB	644	232	<166	36	<26
1,2,4,5-TeCB	399	410	29	103	7
PentaCB	43	38.8	<3.6	91	<8
Hexa-CB	26	18.5	<2.2	72	<9
alpha-HCH	26	14.5	<2.3	56	<9
gamma-HCH	35	18.6	<2.9	53	<8
Chlorophenols:					
2,6-DCP	631	553	142	88	22.5
2,4-DCP	589	579	137	98	23
3,4-DCP	1193	1047	<26	88	<2
2,4,6-TCP	281	272	<4.4	97	<1.5
2,3,4,5-TeCP	130	91	<1.9	70	<1.5
Penta-CP**	74	16.5	5	22	7

** Pentachlorophenol was found in the injection port.

TABLE A18

Collection Experiment #18 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1442min Flow:.255 m^3/min Total Volume: 367m^3 Temp:21-23°C

Prefilter: clean charcoal

Trap 1: PUF/ 16 g XAD-2/ PUF

Trap 2: PUF/ 16 g XAD-2/ PUF

Compound	ng Injected	ng Found Trap 1	ng Found Trap 2	% of injected Trap 1	% of injected Trap 2
Organochlorines:					
m-DCB	3519	231	697	7	20
p-DCB	3722	2139	590	57	16
o-DCB	2627	673	345	26	13
1,2,4-TCB	555	226	<285	41	<51
1,2,4,5-TeCB	344	243	23.6	71	7
PentaCB	37	23.9	<7.6	65	<21
Hexa-CB	22	11.3	<3.7	51	<17
alpha-HCH	22	8.5	<11.4	38	<51
gamma-HCH	30	14.6	<5.8	49	<19
Chlorophenols:					
2,6-DCP	672	317	131	47	19.5
2,4-DCP	628	425	76	68	12
3,4-DCP	1270	801	24	63	2
2,4,6-TCP	299	72	5	24	2
2,3,4,5-TeCP	139	33	2.6	24	2
Penta-CP	78	4	4	5	5

TABLE A19

Collection Experiment #19 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1438 min Flow:.246m^3/min Total Volume: 354m^3 Temp: 21-24°C

Prefilter: charcoal (1 previous use)

Trap 1: PUF/ 16 g XAD-2/ PUF

Trap 2: PUF/ 16 g XAD-2/ PUF

50 ul of std. 311 was spiked onto the front trap during SB.#2.

Compound	ng injected	ng Found Trap 1	ng Found Trap 2	% of injected Trap 1	% of injected Trap 2
Chlorophenols:					
2,6-DCP	1027	691	41.2	67	4
2,4-DCP	960	735	14.9	77	1.5
3,4-DCP	1942	1842	23.3	95	1.2
2,4,6-TCP	458	360	9.6	79	2.1
2,3,4,5-TeCP	212	230	1.7	108	0.8
Penta-CP	120	31.8	12.1	26	10.1

Organochlorines: The organochlorines were not introduced into the system the resulting system blank is reported in SB #2.

TABLE A20

Collection Experiment #20 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1444min Flow:0.257m^3/min Total Volume: 371m^3 Temp:20-21°C

Prefilter: charcoal

Trap 1: PUF/ 30.0 g Florisil/ PUF Solvent: 15% EE in PET

Trap 2: PUF/ 30.0 g Florisil/ PUF

		ng Found		% of injected	
Compound	ng Injected	Trap 1	Trap 2	Trap 1	Trap 2
Organochlorines:					
m-DCB	3508	<17	<18	<0.5	<0.5
p-DCB	3710	<32	<36	<0.9	<1.0
o-DCB	2619	53	45	2	1.7
1,2,4-TCB	553	106	95	19	17
1,2,4,5-TeCB	343	176	62	51	18
PentaCB	37	26.2	<1	71	<3
Hexa-CB	22	17.4	<0.8	79	<4
alpha-HCH	22	27.7	3.1	125	14
gamma-HCH	30	17.4	2.2	58	7.3
Chlorophenols:					
2,6-DCP	624	162	7.1	26	1
2,4-DCP	584	25	77	4.3	13
3,4-DCP	1181	142	<1.2	12	<0.1
2,4,6-TCP	278	44.6	<0.6	16	<0.2
2,3,4,5-TeCP	129	9.8	<0.3	7.6	<0.2
Penta-CP	73	2.6	2.0	3.6	2.7

TABLE A21

Collection Experiment #21 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time:1443min Flow:0.249m^3/min Total Volume: 359m^3 Temp:20-22°C

Prefilter: charcoal (straight from jar, no cleanup)

Trap 1: PUF/ 30 g Florisil/ PUF Solvent: 20% EE in PET

Trap 2: PUF/ 30 g Florisil/ PUF

Compound	ng Injected	ng Found Trap 1	ng Found Trap 2	% of injected Trap 1	% of injected Trap 2
Organochlorines:					
m-DCB	3732.0	<21.0	**	-	
p-DCB	3947.0	<51.0	-	-	
o-DCB	2786.0	<28.0	-	-	
1,2,4-TCB	588.7	198.6	-	34	
1,2,4,5-TeCB	364.7	211.0	-	58	
PentaCB	39.1	34.7	-	89	
Hexa-CB	23.5	18.1	-	77	
alpha-HCH	23.5	15.4	-	66	
gamma-HCH	31.9	16.4	-	51	
Chlorophenols:					
2,6-DCP	647.0	397.0	-	61	
2,4-DCP	604.8	245.0	-	41	
3,4-DCP	1223.8	807.7	-	66	
2,4,6-TCP	288.5	147.1	-	51	
2,3,4,5-TeCP	133.9	48.8	-	36	
Penta-CP	75.6	11.8	-	16	

** Trap #2 was lost during the workup.

TABLE A22

Collection Experiment #22 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1434min Flow:0.243m^3/min Total Volume: 348m^3 Temp:22-22°C

Prefilter: charcoal

Trap 1: PUF/ 16 g XAD-2/ PUF

Trap 2: PUF/ 16 g XAD-2/ PUF

Compound	ng Injected	ng Found Trap 1	ng Found Trap 2	% of injected Trap 1	% of injected Trap 2
Organochlorines:					
m-DCB	3679	914	1100	25	30
p-DCB	3891	1085	1336	28	34
o-DCB	2746	2103	4804	77	175
1,2,4-TCB	580	306	<256	53	<44
1,2,4,5-TeCB	359.5	310	65	86	18
PentaCB	38.5	30.7	<6.4	80	<17
Hexa-CB	23.1	18.1	<5.5	78	<24
alpha-HCH	23.1	13.4	<4.1	58	<18
gamma-HCH	31.5	18.3	<5.2	58	<16
Chlorophenols:					
2,6-DCP	659	347	21.7	53	3
2,4-DCP	616	97.6	8.0	16	1
3,4-DCP	1247	312	1.3	25	0.1
2,4,6-TCP	294	103	0.59	35	0.2
2,3,4,5-TeCP	136	31.4	0.63	23	0.5
Penta-CP	77	3.9	1.8	5	2

TABLE A23

Collection Experiment #23 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1390 min Flow: 0.241 m^3/min Total Volume: 334.4 m^3 Temp: 21-23°C

Prefilter: 230 g sodium thiosulfate/ 1 cm PUF/ ~110 g uncleaned charcoal / 1 cm PUF

Trap 1: PUF/ 5 g Tenax-GC/ PUF

Trap 2: PUF/ 5 g Tenax-GC/ PUF

Compound	ng Injected	ng Found[a] Trap 1	 Trap 2	% of injected Trap 1	 Trap 2
Organochlorines:					
m-DCB	3444	<30	<203	<0.9	<6
p-DCB	3643	<70	<262	<2	<7
o-DCB	2571	<36	<150	<1	<6
1,2,4-TCB	543	202	159	38	29
1,2,4,5-TeCB	337	235	<4.7	70	<1
PentaCB	36.1	19.8	<5.3	55	<15
Hexa-CB	21.7	10.4	<2.7	48	<12
alpha-HCH	21.7	6.4	<7.2	29	<33
gamma-HCH	29.5	11.5	<2.7	39	<9
Chlorophenols:					
2,6-DCP	587	587	54.4	100	9
2,4-DCP	549	541	76.5	98	14
3,4-DCP	1111	1001	<8.9	111	<0.8
2,4,6-TCP	262	228	5.2	87	2
2,3,4,5-TeCP	121.5	115	2.2	95	2
Penta-CP	68.6	25	1.1	37	2

---

a.) corrected for average operational blank in case of organochlorines, but not in case of chlorophenols

TABLE A24

Collection Experiment #24 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1399 min Flow:0.262m^3/min Total Volume: 366m^3 Temp:20.4-22°C

Prefilter: charcoal (no solvent cleanup)

Trap 1: PUF/ 30g Florisil/ PUF

Trap 2: PUF/ 30g Florisil/ PUF

Compound	ng Injected	ng Found Trap 1	 Trap 2	% of injected Trap 1	 Trap 2
Chlorophenols:					
2,6-DCP	682	159	261	23	38
2,4-DCP	637	7.0	58	1	9
3,4-DCP	1290	954	26	74	2
2,4,6-TCP	304	183	9.1	60	3
2,3,4,5-TeCP	141	81	2.4	58	2
Penta-CP	80	33.5	20	42	25

Organochlorines:

The organochlorines were not reported for this experiment.

TABLE A25

Collection Experiment #25 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1444 min Flow:0.257m^3/min Total Volume: 371m^3 Temp: 18-21°C

Prefilter: new Fisher charcoal (uncleaned)

Trap 1: PUF/ 30 g Florisil/ PUF Solvent: 20% EE in PET

Trap 2: PUF/ 30 g Florisil/ PUF

Compound	ng Injected	ng Found Trap 1	 Trap 2	% of injected uncorrected Trap 1	 Trap 2
Organochlorines:					
m-DCB	3504	236	<48	7	<1.4
p-DCB	3706	261	<123	7	<3.3
o-DCB	2616	804	405	31	15
1,2,4-TCB	553	251	236	45	43
1,2,4,5-TeCB	342	160	58	47	17
PentaCB	36.7	26.6	3.6	72	10
Hexa-CB	22.1	18.0	6.5	82	21
alpha-HCH	30.6	23.8	2.5	78	11
gamma-HCH	30.0	21.3	1.4	71	5

Chlorophenols:

The chlorophenols were not reported for this experiment.

TABLE A26

Collection Experiment #26 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1433min Flow:0.219m^3/min Total Volume: 314m^3 Temp:19-19°C

Prefilter: new Fisher charcoal (no cleanup)

Trap 1: PUF/ 5 g Tenax-GC/ PUF

Trap 2: PUF/ 5 g Tenax-GC/ PUF

Compound	ng Injected	ng Found Trap 1	 Trap 2	% of injected Trap 1	 Trap 2
Organochlorines: The OC's were not spiked into the system.					
m-DCB	0.0	289 *	345	-	-
p-DCB	0.0	600.5*	855	-	-
o-DCB	0.0	832 *	1311 *	-	-
1,2,4-TCB	0.0	190	306	-	-
1,2,4,5-TeCB	0.0	29 *	62.7*	-	-
PentaCB	0.0	2.5	8.2	-	-
Hexa-CB	0.0	1.4*	3.7	-	-
alpha-HCH	0.0	4.45	12.8	-	-
gamma-HCH	0.0	2.5	4.2	-	-
Chlorophenols:					
2,6-DCP	665	361	26	54	4
2,4-DCP	622	369	41	59	7
3,4-DCP	1259	390	3.8	31	0.3
2,4,6-TCP	297	77	2.1	26	0.7
2,3,4,5-TeCP	138	22	2.1	16	1.5
Penta-CP	77.8	3.5	4.3	4.5	6

---

*Compound was hard to identify (peak outside of retention time window).

TABLE A27

Collection Experiment #27 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1441min Flow:0.198m^3/min Total Volume: 285m^3 Temp:19-19.6°C

Prefilter: sodium thiosulfate/ charcoal

Trap 1: PUF/ 5 g Tenax-GC/ PUF

Trap 2: PUF/ 5 g Tenax-GC/ PUF

Compound	ng Injected	ng Found[a]		% of injected	
		Trap 1	Trap 2	Trap 1	Trap 2
Organochlorines:					
m-DCB	3394	743	506	22	15
p-DCB	3589	767	719	21	20
o-DCB	2534	712	726	28	29
1,2,4-TCB	535	451	144	84	27
1,2,4,5-TeCB	332	289	<28	87	<8
PentaCB	35.5	30.9	<4.2	87	<12
Hexa-CB	21.4	16.3	<1.8	76	<8
alpha-HCH	29.7	23.4	<8.4	79	<28
gamma-HCH	29.0	24.6	<3.8	85	<13
Chlorophenols:					
2,6-DCP	665	296	30	44	4
2,4-DCP	622	300	31.6	48	5
3,4-DCP	1259	617	75.5	49	6
2,4,5-TCP	297	71.3	3.0	24	1
2,3,4,5-TeCP	138	37.7	2.2	27	2
Penta-CP	77.8	4.1	3.6	5	5

a) corrected for average operational blank in case of organochlorines, but not in case of chlorophenols

TABLE A28

Collection Experiment #28 PUF-Adsorbent Sandwich in PS-1 Sampler

Collection Data

Time: 1392min Flow:0.207m^3/min Total Volume: 288m^3 Temp:19-20°C

Prefilter: solvent cleaned charcoal

Trap 1: PUF/ 6.72 g Tenax-GC/ PUF

Trap 2: 3 g Tenax-GC/ PUF

Compound	ng Injected	ng Found Trap 1	 Trap 2	 Filter	% of injected T 1	 T 2	 Filter
Organochlorines: The chlorobenzenes were not spiked into the system.							
m-DCB	0.0	876	-	-			
p-DCB	0.0	1456	-	-			
o-DCB	0.0	196	-	-			
1,2,4-TCB	0.0	159	-	-			
1,2,4,5-TeCB	0.0	8.2	-	-			
PentaCB	0.0	1.8	-	-			
Hexa-CB	0.0	2.1	-	-			
alpha-HCH	0.0	4.5	-	-			
gamma-HCH	0.0	1.4	-	-			
Chlorophenols:							
2,6-DCP	513	441	7.2	<3.0	86	1.4	-
2,4-DCP	480	401	4.3	<3.0	84	0.1	-
3,4-DCP	971	835	<1.9	<3.0	86	<0.2	-
2,4,6-TCP	229	169	<0.2	0.7	73	<0.1	0.3
2,3,4,5-TeCP	106	78	<.2	1.6	74	<0.2	1.5
Penta-CP	60	28	15.2	480	47	25	800?

TABLE A29

KEY

EXP. #
(air volume m^3)
ADSORBENT
Front/Back
XXg/XXg

RECOVERY	
Front %	XX
Back %	XX
Total %	XXX

COLLECTION EXP FOR PUF/TENAX-GC

	CE #13 (346) 10g/3g	CE #14 (358) 5g/5g	CE #15 (353) 5g/5g	CE #23 (334) 5g/5g	CE #27 (285) 5g/5g
METADICHLOROBENZENE	<27 <20 -	<16 <16 -	<13 <13 -	<0.9 <6 -	22 15 37
PARADICHLOROBENZENE	<47 <40 -	<29 104 104	<24 <24 -	<2 <7 -	21 20 41
ORTHODICHLOROBENZENE	<35 <30 -	<25 155 155	<18 <18 -	<1 <6 -	28 29 57
1,2,4-TRICHLOBENZENE	<78 <25 -	102 <39 102	40 <19 40	38 29 67	84 27 111
1,2,4,5-TETRACHLOROBENZENE	89 <40 89	104 <29 113	88 <25 88	70 <1 70	87 <8 87
PENTACHLOROBENZENE	93 <24 93	102 <15 102	99 <15 99	55 <15 55	87 <12 87
HEXACHLOROBENZENE	98 <38 98	109 <25 109	94 <25 94	48 <12 48	76 <8 76
ALPHA-HCH	68 <38 68	79 <2 79	66 <28 66	29 <33 29	79 <28 79
GAMMA-HCH	112 <35 112	83 <21 83	86 <22 86	39 <9 39	85 <13 85

TABLE A30

```
************************
*       KEY            *
*           EXP. #     *
*    (air volume m^3)  *
*         ADSORBENT    *
*        Front/Back    *
*          XXg/XXg     *
* RECOVERY             *
*  Front % |   XX  |   *
*   Back % |   XX  |   *
*  Total % |__XXX__|   *
************************
```

COLLECTION EXP FOR PUF/ TENAX-GC

	CE #13 (346) 10g/ 3g	CE #14 (358) 5g/ 5g	CE #15 (353) 5g/ 5g	CE #23 (334) 5g/ 5g	CE #26 (314) 5g/ 5g	CE #27 (285) 5g/ 5g	CE #28 (288) 7g/ 3g
2,6-DCP	106 <22 106	79 15 94	94 11 105	100 9 109	54 4 58	44 4 48	86 1 87
2,4-DCP	149 <29 149	98 11 109	118 <11 118	98 14 112	59 7 66	48 5 53	84 0.1 84.1
3,4-DCP	116 <11 116	85 <4 85	100 <4 100	111 <1 111	31 0.3 31.3	49 6 55	86 <0.2 86
2,4,6-TCP	106 <2 106	90 <8 90	95 <4 95	87 2 89	26 1 27	24 1 25	73 <0.1 73
2,3,4,5-TeCP	65 <10 65	63 3 66	85 <3 85	95 2 97	16 2 18	27 2 29	74 <0.2 74
PENTA-CP	35 30 65	18 900 918	34 5 39	37 2 39	5 6 11	5 5 10	- - -

TABLE A31

KEY

EXP. #
(air volume m^3)
g adsorbent
Front/Back
XXg/XXg

RECOVERY	
Front %	XX
Back %	XX
Total %	XXX

COLLECTION EXP FOR PUF/XAD-2

	CE #16 (372) 14g/14g	CE #17 (372) 14g/14g	CE #18 (367) 16g/16g	CE #22 (348) 16g/16g
METADICHLOROBENZENE	<5 <5 -	14 6 20	7 20 27	25 30 55
PARADICHLOROBENZENE	12 275 287	52 33 85	57 16 73	28 34 62
ORTHODICHLOROBENZENE	<7 <7 -	164 28 192	26 13 39	77 175 252
1,2,4-TRICHLOROBENZENE	<76 <55 -	36 <26 36	41 <51 41	53 <44 53
1,2,4,5-TETRACHLOROBENZENE	118 14 132	103 7 110	71 7 78	86 18 104
PENTACHLOROBENZENE	149 18 167	91 <8 91	65 <21 65	80 <17 80
HEXACHLOROBENZENE	132 21 153	72 <9 72	51 <17 51	78 <24 78
ALPHA-HCH	102 <59 102	56 <9 56	38 <51 38	58 <18 58
GAMMA-HCH	109 <23 109	53 <8 53	49 <19 49	58 <16 58

TABLE A32

KEY
EXP. #
(air volume m^3)
ADSORBENT
Front/Back
XXg/XXg
RECOVERY
Front % XX
Back % XX
Total % XXX

COLLECTION EXP FOR PUF/ XAD-2

	CE #16 (372) 14g/14g	CE #17 (372) 14g/14g	CE #18 (368) 16g/16g	CE #19 (354) 16g/16g	CE #22 (348) 16g/16g
2,6-DICHLORO-PHENOL	71 8 79	88 22 110	47 19 66	67 4 71	53 3 56
2,4-DICHLORO-PHENOL	77 13 90	98 23 121	68 12 80	77 1 78	16 1 17
3,4-DICHLORO-PHENOL	75 <3 75	88 <2 88	63 2 65	95 1 96	25 0.1 25
2,4,6-TRICHLO-PHENOL	69 <2 69	97 <2 97	24 2 26	79 2 81	35 0.2 35
2,3,4,5-TETRA-CHLOROPHENOL	35 <2 35	70 <2 70	24 2 26	108 1 109	23 1 24
PENTACHLORO-PHENOL	19 7 26	22 7 29	5 5 10	26 10 36	5 2 7

TABLE A33

KEY
EXP. #
(air volume m^3)
ADSORBENT
Front/Back
XXg/XXg
RECOVERY
Front % | XX
Back % | XX
Total % | XXX

COLLECTION EXP FOR PUF/ FLORISIL

	CE #20 (371) 30g/30g	CE #21 (359) 30g/30g
METADICHLOROBENZENE	<0.5 <0.5 -	<0.6 * -
PARADICHLOROBENZENE	<1 <1 -	<1.3 - -
ORTHODICHLOROBENZENE	2 2 4	<1 - -
1,2,4-TRICHLOROBENZENE	19 17 36	34 - -
1,2,4,5-TETRACHLOROBENZENE	51 18 69	58 - -
PENTACHLOROBENZENE	71 <3 71	89 - -
HEXACHLOROBENZENE	79 <4 79	77 - -
ALPHA-HCH	125 14 139	66 - -
GAMMA-HCH	58 7 65	51 - -

*Back trap was lost in workup.

TABLE A34

KEY

EXP. #
(air volume m^3)
ADSORBENT
Front/Back
XXg/XXg

RECOVERY	
Front %	XX
Back %	XX
Total %	XXX

COLLECTION EXP FOR PUF/ FLORISIL

	CE #20 (371) 30g/30g	CE #21 (359) 30g/30g
2,6-DICHLORO-PHENOL	26 1 27	61 @ -
2,4-DICHLORO-PHENOL	4 13 17	41 - -
3,4-DICHLORO-PHENOL	12 <0.1 12	66 - -
2,4,6-TRICHLO-PHENOL	16 <0.2 16	51 - -
2,3,4,5-TETRA-CHLOROPHENOL	8 <0.2 8	36 - -
PENTACHLORO-PHENOL	4 3 7	16 - -

[@] Back trap was lost in workup.

TABLE A35

COLLECTION EXP FOR TENAX IN STACKED SIEVES

	CE #1 (100)	CE #2 (100)
PARADICHLOROBENZENE	-	-
	-	-
	-	-
	-	-
	-	-
ORTHODICHLOROBENZENE	-	21.7
	-	17.4
	-	8.2
	-	7.5
	-	54.8
1,2,4-TRICHLOROBENZENE	-	59.1
	-	8.1
	-	-
	-	-
	-	67.2
HEXACHLOROBENZENE	-	82.2
	-	2.7
	-	0.8
	-	0.8
	-	86.5
NAPTHALENE	70.2	-
	19.9	-
	2.3	-
	<1	-
	92.4	-
2-METHYLNAPTHALENE	82.3	-
	14.8	-
	<2	-
	<2	-
	97.1	-
BIPHENYL	89.4	-
	3.7	-
	<2	-
	<2	-
	93.1	-
2,6-DMN	87.5	-
	7.1	-
	<2	-
	<2	-
	94.6	-

KEY

EXP #
AIR VOLUME M^3

% RECOVERY
TRAP 1
TRAP 2
TRAP 3
TRAP 4
TOTAL

TABLE A36

COLLECTION EXP FOR PUF-TENAX TRAPS IN PS-1 SAMPLER

	CE #3 (37)	CE #4 (34)	CE #5 (32)	CE #6 (119)	CE #7 (117)	CE #8 (165)	CE #9 (78)
P-DCB	1	-	-	-	-	-	-
	81	-	84	-	67	67	54
	-	-	-	-	2	0.6	-
	3	-	3	-	5	8	<0.5
	85	-	87	-	74	76	54
O-DCB	1	-	-	-	-	1	10
	75	-	84	-	68	75	78
	-	-	-	-	<0.7	0.6	-
	<0.2	-	4	-	8	9	6
	76	-	88	-	76	86	94
1,2,4-TRICB	<0.7	-	-	-	-	2	5
	107	-	106	-	94	110	106
	-	-	-	-	<4	<0.3	-
	<0.7	-	0.3	-	0.8	1	-
	107	-	106	-	95	113	111
HCB	26	-	-	-	-	6	<4
	99	-	76	-	98	94	106
	-	-	-	-	<0.9	0.7	-
	<3	-	1	-	<0.9	0.7	-
	125	-	77	-	98	101	106
NAP	-	-	-	1	2	0.8	3
	79	95	-	89	158	76	84
	-	1	-	-	1	0.2	0.2
	0.3	0.5	-	1	14	2	1
	79	96	-	91	175	79	88
2-MN	-	-	-	2	0.6	2	6
	82	97	-	91	91	79	74
	-	-	-	-	-	-	-
	<0.5	<1	-	-	-	-	1
	82	97	-	93	92	79	81
BIPH	-	0.8	-	5	5	5	10
	83	97	-	89	92	75	68
	-	-	-	-	-	-	-
	<0.4	<0.8	-	-	-	-	-
	83	98	-	94	97	80	78
2,6-DMN	-	0.3	-	3	3	3	9
	83	98	-	91	95	78	69
	-	-	-	-	-	-	<0.1
	<0.4	<0.6	-	-	-	-	<0.1
	83	98	-	94	98	81	78

KEY

Exp. #
(air volume) m^3
% Recovery
Trap 1
Trap 2
Trap 3
Trap 4

Trap #5 wasn't determined for these exp.

TABLE A37

```
*************************
*      KEY              *
*           EXP. #      *
*      (air volume m3)  *
*          ADSORBENT    *
*         Front/Back    *
*           XXg/XXg     *
* RECOVERY              *
*  Front %  |  XX  |    *
*   Back %  |  XX  |    *
*  Total %  |  XXX |    *
*************************
```

COLLECTION EXP FOR PUF/ Tenax SANDWICH IN PS-1 SAMPLER

	CE #10 (102) 10g/ 3g	CE #11 (125) 10g/ 3g
PARADICHLOROBENZENE	72 4 76	59 5 64
ORTHODICHLOROBENZENE	76 5 81	62 9 71
1,2,4-TRICHLOBENZENE	83 12 95	91 0.6 92
HEXACHLOROBENZENE	80 - 80	93 - 93
NAPTHALENE	72 2 74	76 2 78
2-METHLYNAPTHALENE	76 0.5 77	83 2 85
BIPHENYL	85 0.3 85	80 - 80
2,6-DMN	87 1 88	82 0.7 83

CE #12 was an operational blank and % recovery was not determined. (see Table A12)